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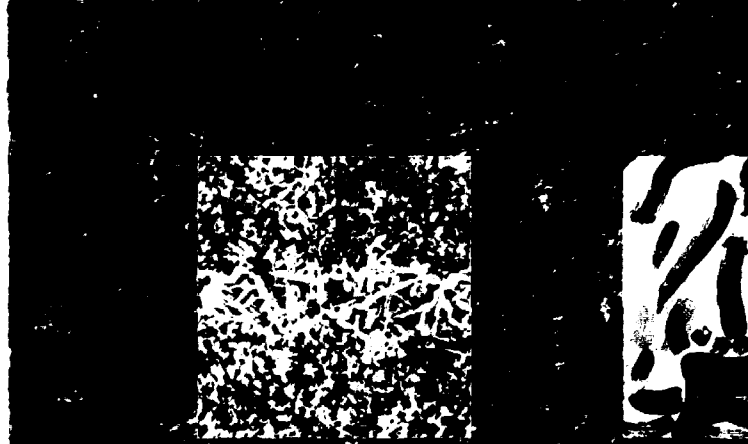
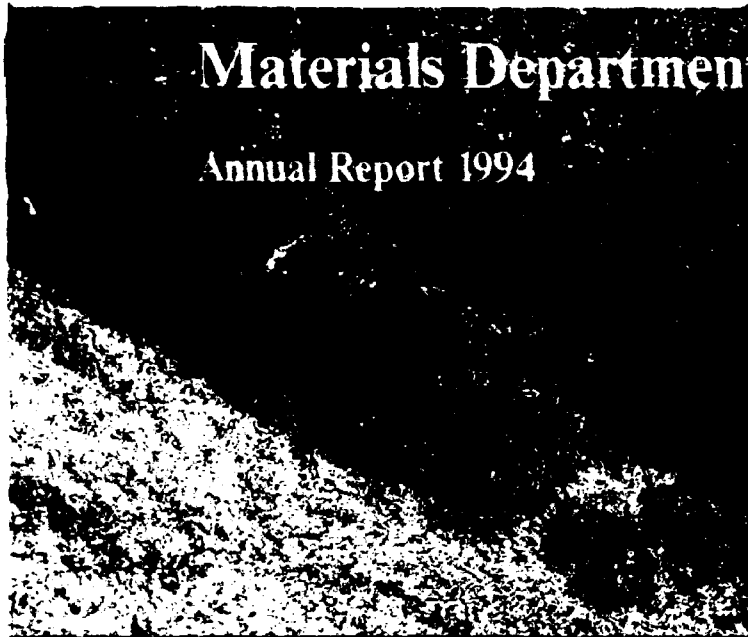
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Annual Report 1994

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Abstract

Selected activities of the Materials Department at Riso National Laboratory during 1994 are described. The work is presented in three main chapters: Materials Science, Materials Engineering and Materials Technology. A survey is given of the Department's participation in international collaboration and of its activities within education and training. Furthermore, the main figures outlining the funding and expenditure of the Department are given. Lists of staff members, visiting scientists, publications, lectures and poster presentations are included.

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At the Materials Department, paintings, lithographs and sculptures decorate meeting rooms and offices as well as corridors, laboratory areas and workshops. Photos of some of these works of art are to be found at various places within this report.

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Introduction – Materials Department 1994



Scanning electron micrograph of a material surface.

European and Danish interest in materials research remained high in 1994 and funding was available both for basic and applied research. Research priorities have, however, changed somewhat. In Denmark, public funding of industry related materials research has decreased and as a result, the collaborative work between the Department and Danish industry on advanced materials and processes has been reduced. Fortunately, it has been possible to replace the lost funding through increased participation in energy programmes, especially those related to solid oxide fuels cells, wind energy and bio-fuel combustion. These programmes, partly sponsored by the Ministry of Energy, are carried out in collaboration with Danish utilities and Danish industry and links with new partners have therefore been established.

The change in sources of funding and, in consequence, of research objectives has required a high degree of flexibility within the Department's research and administration. This flexibility has been achieved as a result of the experience of the staff which has been acquired through our involvement over the years in many large national and international research programmes. It has also been important to have sufficient resources to hire new staff and to make investments in advanced scientific and technical equipment. Finally, flexibility has been achieved by putting more responsibility on our senior researchers, not only with regard to innovative research, but also to ensure external funding for their research.

Research strategy

The research strategy of Riso and of the Materials Department is linked to the Danish national policy for research and development. In 1994 a new government was formed and the new Ministry for Research, created in 1993, had not had time to up-date national research strategy. Input to updating of research policy is, however, now appearing, being partly based on a recent OECD evaluation of Danish research. This evaluation was followed by hearings with broad participation from within Danish society. This input has allowed some political guidelines to be formulated which emphasize the following:

- Over the next 5 years the Government plans to increase funding for research and development to 2% of the GNP. This means an increase in public funded research by DKK 500 million per year (USD 85 million), equivalent to approximately 15% increase over current R&D spending.
- It is acknowledged that research policy is linked to industry and trade policy and it is emphasised that the results of research must find application in industry with the aim of creating new jobs.
- Increased funding for public research will only be given in areas where researchers and research teams are of international standard. Other re-

search areas will be maintained at their present level. A national evaluation of research areas and research groups is therefore currently planned to be carried out in 1995.

- Finally, it has been confirmed that the overall energy policy will be continued and that the energy research programme shall aim at fulfilling political goals such as the reduction in overall energy consumption and in carbon dioxide emissions.

These issues are to a large extent covered in the contract from 1993 between the Ministry of Research and Technology (now the Ministry of Research) and Riso National Laboratory. This contract of four years' duration ensures that Riso's budget is to be exempt from annual cuts. In return, Riso has undertaken to complete a number of predefined research tasks (based on a strategic plan "Riso 2000"), streamline its administration and perform a poll among users at the beginning and at the end of the four year period. It should, however, be noted that the budget which is to be exempt from cuts in this contract is the basic government funding, this being about 55% of Riso's total income. The remainder stems from many different sources and is most frequently acquired in competition with other research laboratories. It is therefore essential that changes in trends for external research funding are followed carefully in order to maintain, or hopefully increase, the current level of activity. It is also very important to maintain good contacts with the users and in this context, it has been satisfying that an enquiry carried out in 1994 was generally rather positive for Riso.

For the Materials Department, a follow-up strategy requires that research must continue to be high level. It is also very important that this research is acknowledged at the national level both for being of international quality and for its relevance to the Danish society. This is a prerequisite for maintaining national funding in the future. In terms of quality, our impression is that the work of the Materials Department is acknowledged internationally as being at the forefront of materials research and development. That the research carried out is of relevance to society is illustrated by our many contacts and formal contracts with Danish and European industry and by the very active participation of the Department in energy programmes sponsored by the Danish utilities and the Ministry of Energy. This indication was reinforced by the many positive replies about the Department which came out of the enquiry among users mentioned above. Not wanting to rest on our laurels however, many new links are currently being created both with research groups, industry and teaching institutions at home and abroad. With this in mind, a number of new initiatives have been taken in 1994 and are reported in the following sections.

Resources

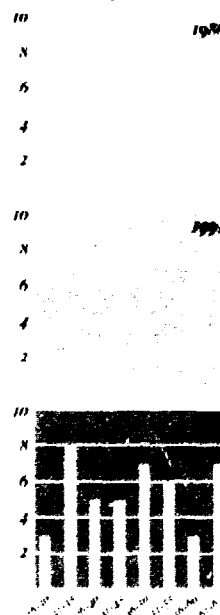
The income of the Materials Department has been satisfactory in 1994, being at approximately the same level as in 1993. This, together with capital from previous years, has allowed DKK 3 million (~USD 0.5 million) to be invested in equipment and a further 4.5 million (~USD 0.8 million) to be reserved for refurbishment of the hot cell building in 1994/95. This re-modelling and building

will cost about DKK 24 million (~USD 4.1 million), requiring DKK 19.5 million (~USD 3.3 million) from a combination of Riso's central funds and from a special allocation from the government.

The maintenance of the 1994 income at the 1993 level has been achieved under somewhat difficult conditions. By comparison with 1993, there has been a major change in the balance of industrially related and basic research. Public funding of the Department's industry related research fell from 25% to 10% of the total income while funding for basic research and energy research increased from 20% to about 25% and 30%, respectively. Finally, due to a late start of the Fourth Framework Programme within EC, funding from this source has decreased from 15% to 10% in 1994.

The present staff situation, in which the Department has been able to sustain a moderate level of controlled growth by hiring young engineers, scientists and technicians, continues to be satisfactory. This has made it possible to maintain a broad distribution in ages, particularly among the research staff. The influx of post graduate students has increased markedly.

Academic staff, age distribution



mainly due to the creation of the Engineering Science Centre. A consequence of the increase in post graduate teaching has been an increase in collaboration with the university departments with which we jointly supervise the post graduate students.

New programmes and a steady increase in staff numbers in recent years has put considerable pressure on existing facilities. In October 1994, the decommissioning of the hot cells was completed and most of the interior of the buildings demolished. Refurbishment was started at the beginning of 1995 and new laboratories and offices will be ready in the Autumn of 1995. In total, about 2000 square meters will be available to house activities such as fuel cell research, ceramic processing, powder metallurgy and polymer composite technology. There will also be space for new educational facilities directed towards high schools, universities and industrial companies. Facilities will also be created in order to introduce information technology into both the teaching and research activities.

Organization

The research at Riso is organized in programme areas, programmes and projects. Within the Materials Department the project work is carried out within the three research areas or disciplines:

- Materials Science (theory and characterization).
- Materials Engineering (modelling and performance).
- Materials Technology (synthesis, processing and products).

Research and Development

Materials Science
Materials Engineering
Materials Technology
Energy Materials

Market and Customers

Research and Education *Energy Sector* *Industry Sector*

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The combination of these disciplines within the Department makes it possible to carry out work both in the development, characterization and modelling of materials and processes and on the application of materials in advanced products. This combination of disciplines under one roof is unique in Denmark and has allowed the Department to acquire complex projects which require very specialised knowledge. We have also been able to launch large programmes based on an integration of the three disciplines. This organization has therefore made the Department quite flexible in the market place.

The work in the Department is organized in a number of programmes and projects, each headed by a programme or project leader. These leaders are responsible for both the scientific quality and the administration of their programmes and projects. Due to the large number of projects, coordination is imperative. For large projects or groups of related projects, management or coordination groups are established which combine scientific, technological and administrative expertise. Examples of such activities are the Fuel Cell Programme, the Engineering Science Centre and IT contracts in which staff members are programme managers. An important part of the work is

to formulate new projects both for internal and external funding. This is done by the researchers themselves, individually or in groups, who are able to call upon other staff within the Department who are experienced in administration and in marketing. The size of the Department requires that the use and expenditure of all resources be thoroughly followed and monitored. This is done through a group consisting of the management of the Department together with four programme managers. This regular follow-up of the research activities in the Department ensures flexibility in the expenditure of resources and of manpower in particular, combined with an administrative control of income and expenditure.

Research

In this report the work in the Materials Department is described in three chapters, one for each of the research disciplines. Each chapter therefore provides a general description of the work, with emphasis on new experimental findings, models and novel techniques. The description of the work on energy materials (for example, fuel cell projects) which, in organizational terms is treated as a separate research area, is integrated into the individual chapters.

Research programmes related to basic research, energy and industry are summarized below. In this way, the introduction attempts to characterize the Department's contact to markets and customers (industry, energy and research). The individual sections which follow illustrate the research and development work.

Basic research

The applied programmes described below are founded on long term basic programmes covering modelling of materials properties and behaviour as well as characterization of structures and properties. The materials investigated are metals, ceramics and polymers in bulk and in composite materials. The work on composites is still given high priority and the Department is active in programmes on metal matrix composites, polymer composites and ceramic matrix composites. Together with the Technical University of Denmark (DTU), new research activities have been started to study the structure and properties of metallic multi-layers manufactured by electrochemical deposition techniques.

In 1994, research on structural characterization and modelling of materials has been carried out in the Engineering Science Centre at Risø which is financed by the Danish Technical Research Council. This centre carries out long term research and trains post graduate students (about 10) and post doctoral fellows. Complementary to the activities within this centre, a number of projects are concentrated on the themes of metals processing and modelling. The main research areas are numerical and experimental modelling of processes, such as the microstructural and texture

changes which occur during deformation and annealing of materials. Part of this work is included in a new Centre financed by the second 'Materials Development Programme' at P2 for 4 years and with 5 partners from the Technical University of Denmark and Aalborg University. The title of this Centre is Materials Processing, Properties and Modelling (MP2M). The areas covered by this Centre and the Engineering Science Centre are currently in focus both nationally and internationally. This is because it now appears to be within reach to introduce quantitative observations of microstructure, texture, mechanical properties and residual stress into general models of the mechanical and thermal behaviour of metals. This approach is used in a number of programmes directed towards practical applications in collaboration with Danish and international industrial companies.

A natural consequence of the type of research carried out in the Department is that considerable emphasis is placed on the develop-

ment of various experimental techniques. Major areas are the application of neutron scattering and electron microscopy and diffraction. In the area of electron diffraction, automated techniques for crystallographic orientation measurements are now available using electron back scattering patterns (EBSP). For the same type of measurements with a higher spatial resolution, semi-automatic Kikuchi pattern analysis is routinely used. Also, in the area of electron microscopy, a new in-situ technique has been developed together with the Engineering Department, Cambridge University which allows simultaneous mechanical loading and structural observations in the Department's environmental scanning electron microscope.

A new line of research in the Department was initiated in 1994 with the application of high energy X-rays from a synchrotron facility to study nucleation and growth during recrystallization. It appears that this technique will have a large potential in materials research as a

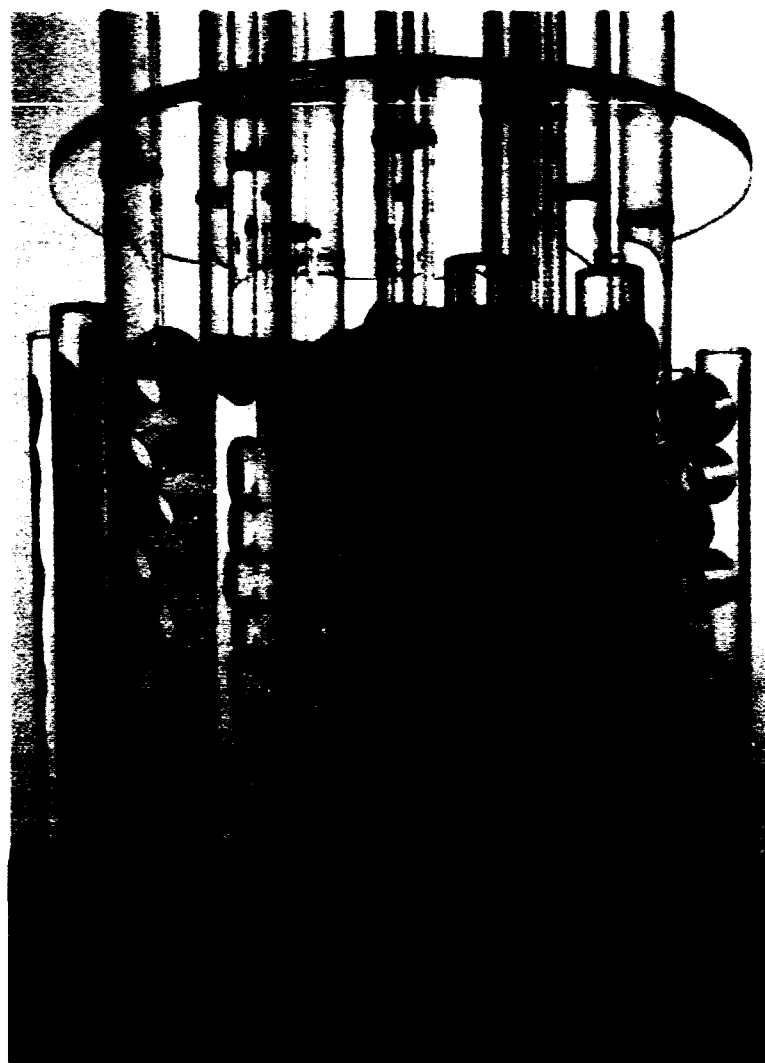


complementary method to neutron and electron diffraction. The technique is particularly suited to the examination of bulk structural characteristics, non-destructively and with high spatial resolution. The synchrotron experiments will be carried out at the Hasylab in Hamburg; future possibilities for experiments at the European Synchrotron Radiation Facility (ESRF) in Grenoble are also being explored.

Energy related research

The primary research activities within this area are related to the development of solid oxide fuel cells (SOFC). This work focuses on the fabrication of small prototype modules and on research to improve the efficiency, economy, durability and versatility of fuel cells. One of the milestones of this programme is to achieve operation of a small prototype (0.5-1 kW) in the Summer of 1995. This work is supported by the ELSAM electricity utility group and by the Ministry of Energy. The overall programme is managed by the Materials Department with the participation of Haldor Topsøe A/S and several research organisations. The Danish programme includes both short- and long-term objectives, the latter being integrated into a number of programmes supported by Nordic organizations and by EC.

Energy research also covers a programme together with Danish industry and the Danish utility groups ELSAM and ELKRAFT on advanced materials for waste and bio-fuel combustion. Participants also include a Danish technological research institute, the Force Institute, and the Technical University of Denmark (DTU); this work is supported by the Ministry of Energy. The aim of the pro-



gramme is to increase the efficiency of electrical production by raising the plant combustion temperature. Achieving this demands the application of many different approaches. The Department, for its part, will concentrate on advanced materials and coatings, materials characterization and materials testing. Further support for this programme will come from participation in COST 501, Work Package 13, 'Clean Combustion Technologies', with leading European industries and research laboratories as participants.

A continuing effort is the materials development, design and testing of materials related to blades for wind turbines. The work in this area has hitherto been part of a JOTI programme (11) with the participation of research institutes and industries in many

European countries, coordinated by the Department. However, a major shift was introduced in 1994 as the Department was asked by the Ministry of Energy together with the Department of Meteorology and Wind Energy at Riso to prepare a joint programme with the participation of utilities and industry. The aim of this programme is to improve the efficiency and profitability of large wind rotors while also considering environmental problems both during manufacturing and during operation of the rotors. A programme was defined which includes design, materials properties, manufacturing technologies and testing of rotors. This project was approved at the end of 1994 for a 3 year period.

Finally, within energy research, the Department is engaged in the

European Fusion Technology programme, encompassing work related to ITER (International Thermonuclear Experimental Reactor). The work in the Department concentrates on studies of irradiation effects in materials and alloys, especially copper and steels. The European Fusion programme is an important part of EU's Fourth Framework Programme allowing the Department's work to be maintained in this area.

Industry related research

A major change in the industry related research followed the replacement of the first Danish Programme on Materials Technology, MT P1, with a continuation programme, MT P2. Collaborative research programmes between industry and research institutions which had been carried out within a number of centres were replaced by newly defined activity areas. Funding was much reduced and in many cases there was a change in research objective. As a result, Departmental activities were



Testing fusion materials

halted within three centres, namely the Centre for Powder Metallurgy, the Centre for Polymer Composites and the Centre for Advanced Technical Ceramics. In the first two centres, new and more focused programmes were obtained in collaboration with Danish industry. However, no funding at all was given to the area of advanced ceramics. The Department still considers ceramics to be potentially very important materials for industrial application due to their unique properties. A number of activities have therefore been maintained, especially within characterization and processing of ceramics. Funding for these activities has been found outside the MT P2 programme.

Another change in industry related research followed modifications in the energy programme. The Ministry of Energy has placed even more emphasis on industrial participation in research and development programmes directed at fulfilling the declared goals of Denmark's energy policy. As one of the results, materials research and technology was given a high priority in programmes on wind energy and bio-fuel combustion and the Department could significantly increase its activities in these areas in collaboration with Danish industry and the utilities.

A third change in industry related research, both at national and international levels, is the increased interest in the characterization and modelling of materials and processes. Such studies have hitherto been regarded as basic research and have been carried out as such in the Department over many years. It should be noted that this interest extends not only to advanced materials but also includes more traditional materials and alloys. A consequence of this trend is an increased collaboration between industrial researchers and

scientists in the Department.

Industrially related research in the Department includes a number of projects carried out on a proprietary basis. Such work relates to design, materials testing, non-destructive testing and failure analysis. Furthermore, the Department undertakes work as an industrial sub-contractor in areas where expertise has been built up, for example in the manufacture of components in polymer composites, processing of ceramics, and dip brazing of electronic parts.

Achievements

A number of scientific and technical achievements during the year may be mentioned.

In the area of Materials Science, studies of local orientation variations in deformed polycrystals have confirmed a theoretical prediction related to the energy of dislocation configurations. Of a more applied nature has been the completion of an industrial MATRI-FCRAM project on hot deformation of aluminium alloys with 11 scientific publications written by 7 partners in 4 countries. For materials characterization, a statistical method has been worked out for the treatment of large amounts of FEM-data and a deformation rig has been installed and applied to in-situ testing of materials in the environmental scanning electron microscope.

In the area of Materials Engineering, experimental observation of the fracture and delamination of metallic multilayers on a ductile substrate has been modelled analytically and by the finite element method (FEM). FEM has also been used to predict strain gradients in metal matrix composites, showing good agreement with structural observations by

transmission electron microscopy. Of a more applied nature, a very light and efficient flywheel has been designed for energy storage. Also, a novel X-ray technique for non-destructive measurement of the wall thickness of tubes has been developed.

In the area of Materials Technology, techniques have been developed for manufacturing industrial components using thermoplastic polymers which are strengthened with glass and/or carbon fibres. In the field of joining, a new method has been developed for brazing powder metallurgy parts with a very limited absorption of the filler alloy. Within the field of solid oxide fuel cells the internal resistance of a cell has been reduced by introducing an ultra-thin intermediate layer between the Ce-Gd anode and the electrolyte.

Finally, the hot cell facility has been fully decommissioned and refurbishment of the building for new laboratory and office space has started.

Hot cell shut-down



International cooperation

The overall Danish research strategy emphasises the need for international cooperation based on international quality of the national research. Within nearly all research areas of the Materials Department there are active international collaborative agreements. This is reflected by the large

number of foreign students and guest scientists who have worked in the Department. Active international cooperation is also demonstrated by the many joint authorships of the scientific publications of the Department.

An important part of international cooperation is the participation in European programmes such as EUREKA, EUCLID, COST. Of special importance is the NUTE-FURAM programme and the NUTE programme where the Department, in 1994, has participated in 7 programmes of which 4 are coordinated by the Department.

These programmes, being part of the 3rd Framework Programme, are now about to be completed. Replacement programmes however, have been delayed due to the general delay in the approval of the 4th Framework Programme. Fortunately, this period of waiting is now over and a number of projects are being formulated for submission in the Spring of 1995.

These proposals are concentrated on themes in which the Department's research record has been successful. Examples are metal forming, microstructural and crystallographic characterization of materials, non-destructive testing of tubes, brazing of powder metallurgical parts, development and durability of solid oxide fuel cell components, design and properties of blades for wind turbines and materials for bio-fuel combustion. A number of these proposals will be coordinated by the Department with additional plans to secure involvement by Danish firms in a number of the projects.

Symposia and workshops

An important activity in 1994 has been the organization of the 15th Riso International Symposium on 'Numerical Predictions of

Deformation Processes and the Behaviour of Real Materials'. The symposium followed the format of the Riso symposia series and was attended by about 95 scientists representing industry, research institutes and universities from around the world. Another activity was the organization of a one-day meeting covering both the activities of the Engineering Science Centre and a review of a major NUTE project on characterization and modelling of various forming processes. This latter project was sponsored by the NUTE programme and was carried out in collaboration with two industrial partners and the Technical University of Denmark. Other meetings involving industry and research institutions in Denmark have been arranged as part of the culmination of the centres under NUTE.

Public relations

RISO activities have again included general media coverage of research and development. There has also been a large number of visitors to the Department. Another activity is the continuation of a series of papers, in Danish, on important research and development activities. Finally, the staff of the Department participated in a well-attended meeting with undergraduate and graduate students to inform them about the possibility of carrying out materials research at Riso.

Education

Educational activities are becoming an increasingly important part of the work of the Department. Many staff members act as external lecturers and examiners at undergraduate and post graduate levels. Also, under-



graduates and graduates from engineering schools in Denmark and abroad have carried out their experimental work and received academic training in the Department, supervised by staff members.

The number of graduate students and post graduates has increased significantly in recent years due to new funds, both nationally and within the EU. About 20 young researchers are currently carrying out their studies in the Department. In addition, the Department teaches apprentices in mechanics and electronics.

Educational activities also include participation in a comprehensive course for technicians and engineers on materials properties, processing, testing and product design. An important part of this programme is to offer courses for in-service training of employees from Danish industry. To widen access and to reduce the cost of such training the Department is planning to develop facilities for so-called 'remote classroom' teaching.

The initiatives taken in 1993, directed towards teaching at the

high school level have now resulted in the preparation, together with local high school teachers, of the first 'trial run' course. This first course, which will cover processing and properties of polymers and polymer composites, will be taught in March 1995. The teaching will consist of lectures held at the school followed by laboratory experiments carried out in the Department.

The many educational and training activities involve a large number of guests, many of them from abroad, staying at the Department. The Department therefore tries to help in supplementing the professional and work activities with social and cultural activities. This is especially important for foreign guests and students. Such activities are not new to the Department but the large number of guests has required a more focused effort than before.

Concluding remarks

Materials research in Denmark has become more integrated in

recent years, due primarily to a significant increase in public and industrial funding. At present however, the situation is somewhat unpredictable as new political strategies are currently being formulated for the country's overall research and development plans. These strategies will be divulged in 1995 with expected announcement of measures to monitor the quality and effectiveness of the ongoing research. It is our hope that these initiatives will provide strategies, plans and funding which will allow the formulation of guidelines for materials research for at least the coming 5 years. Only then will it be possible to maintain and renew current research activities which are internationally competitive. This is a necessary prerequisite in ensuring that trained researchers and advanced techniques are available whenever needed. It is also hoped that national plans will be drafted with clear reference to European research as formulated in EU's framework programmes. Coordination will ensure synergy and both strengthen and expand the links between public and industrial research in Europe. This should lead to a further strengthening and internationalization of Danish research in materials.

Finally, it is foreseen that a long term national research strategy will lead to improved integration of the research and teaching activities at universities with the research and development work carried out by the research laboratories and by the industry. The Materials Department is keen to play an active role with such a national network by contributing a recognised research base, state of the art facilities and a very broad international network.

Materials Science – theory and characterization



Fritz Hugel Sachs

Efforts to improve the inherent properties of materials are based on our ability to characterize, understand and finally to modify microstructure. The research in this area, although of a fundamental nature, is often initiated in response to specific technological and engineering demands for new and improved materials. The research themes in Materials Science are therefore closely related to the applied programmes within the Materials Department. Much of this basic research is carried out in close collaboration with colleagues from universities and government research laboratories around the world. The area was strengthened in 1993 in the Materials Department by the establishment of the 'Engineering Science Centre for Structural Characterization and Modelling of Materials'.

Deformation microstructures and textures

Plastic deformation and texture

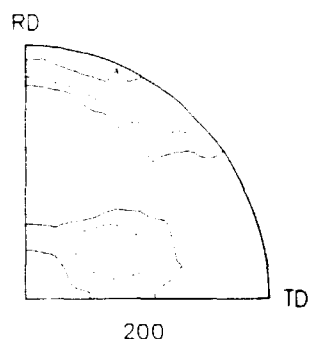
There is a long tradition at the Materials Department for studying the relation between the basic mechanisms of plastic deformation and the resulting deformation texture in metals. There are (at least) two reasons for performing such studies: texture is a technologically important property of materials, and the deformation texture contains important information about the deformation processes.

It is normally assumed that the

basic processes of texture formation (the basic rules for the lattice rotations in the individual grains) are well understood. The problem of understanding texture development is therefore regarded as due to our incomplete understanding of the basic deformation processes. The details of the deformation processes clearly have a profound effect on the resulting deformation textures. For instance, there is no doubt that one of the classic problems in texture research, the difference in rolling texture between fcc materials with high to intermediate stacking fault energies and fcc materials with low stacking fault energies (the difference between the copper-type and the brass-type texture), is related to a difference in deformation mechanism. One possibility is the difference between a Taylor-type deformation pattern for materials with higher stacking fault energies and a 'modified-Sachs' deformation pattern for materials with low stacking fault energies.

However, recent work at the Materials Department (as described in the 1993 annual report) has demonstrated that texture formation during plastic deformation with grain subdivision must follow a set of rules different from the normal simple rules. This has drawn our attention to the fact that there is a substantial uncertainty about the calculation of the lattice rotations associated with a given deformation process (unless deformation follows the simple Taylor mechanism).

This uncertainty is demonstrated in the two figures showing



{200} pole figures for 50% rolling simulated with different rules for the lattice rotations. The simulation at the top corresponds to brass-type texture, while the simulation at the bottom is closer to the copper-type.

simulated fcc {200} pole figures for rolling deformation to 50% reduction according to the modified Sachs model. The difference between the two pole figures stems from the use of different rules for the calculation of the lattice rotations. The pole figure at the top corresponds to a brass-type texture (it is the type of simulated pole figure quoted to substantiate the connection between the brass-type texture and the modified Sachs model mentioned above). The pole figure at the bottom is closer to a copper-type texture. It should be underlined that the difference between the two pole figures is not taken to reflect the physical difference between the formation of the copper-type and the brass-type texture. At the present stage the only conclusion

to be drawn is that different rules for the calculation of the lattice rotations may produce rather different simulated textures.

Microstructures of deformed materials

The microstructure of a material gives information about its strength and about its behaviour during heat treatments. Computerized microstructural techniques provide large amounts of data required for the analysis and modelling of microstructures. Modelling of deformation and heat treatment is then able to advance the processing and properties of metals and alloys. An important part of the microstructural characterization is to study dislocations which have participated in plastic deformation. Part of these dislocations are stored in boundaries in a two or three dimensional arrangement. These boundaries have a significant effect on the mechanical and thermal behaviour of deformed materials. Dislocation boundaries, both in single and polycrystalline materials, have been analyzed using advanced automated tech-

niques developed in the Materials Department; electron-back-scattering pattern (EBSP) techniques in a scanning electron microscope and Kikuchi-analyses in a transmission electron microscope. This analysis has led to an important distinction between two different types of dislocation boundaries. This distinction has made it possible to better formulate the relationship between the strength of a material and the structural parameters. It has also been possible to relate directional properties (anisotropy) to microstructure and to further the understanding of the behaviour of deformed metals during annealing treatments.

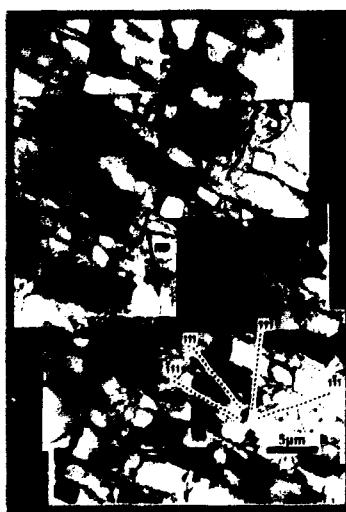
Recrystallization studies

Orientation aspects of recrystallization

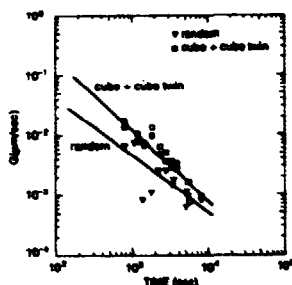
A new trend in recrystallization studies is to combine structural and textural information to obtain a better understanding of the basic processes involved: nucleation and growth.

With new automatic 'local texture techniques' it is possible to determine the crystallographic orientation in selected local areas of the microstructure quickly and accurately. For example, with the Riso automatic electron back scattering pattern (EBSP) technique, the orientation within areas of 1 micron diameter can be determined automatically in about 10 seconds. This technique has been used to determine growth rates during recrystallization and for the first time it has been possible to determine such growth rates for nuclei/grains of the different crystallographic orientations.

For several heavily cold rolled fcc metals the growth rate of cube, rolling and 'randomly' oriented nuclei/grains have been measured.



Transmission electron micrograph showing dislocation arrangement in deformed aluminium. Elongated dislocation cell structure after 5% deformation by cold-rolling



Growth rates of cube, rolling and 'randomly' oriented nuclei/grains have been measured for recrystallization of heavily cold-rolled fcc metals. Cube grains always grow faster than other grains.

In all the cases studied it was found that the cube grains grow faster than the other grains: an example is shown in the figure. This leads to stronger cube textures and a broader grain size distribution than would otherwise have developed and is thus very important for the properties of the recrystallized material.

Faster growth of cube grains has often been ascribed to an optimal misorientation ($40^\circ \langle 111 \rangle$) of the grain to the neighbouring deformed matrix material. Using the EBSP technique it is also possible to get information about the distributions of orientations in the deformed matrix and the misorientation relationships between a growing grain and the surrounding deformed matrix. The results show that the deformed matrix, after the heavy deformation, is broken up into cell blocks which typically have very different orientations. As a consequence, the recrystallization nuclei/grains will be surrounded by matrix material of different orientations at various positions along its periphery. This will change further during its growth as shown in the sketch. The misorientation distribution between the nuclei/

grains and the surrounding matrix is therefore expected to be broad in heavily cold deformed polycrystalline metals, and not a sharp, for example $40^\circ \langle 111 \rangle$, rotation. This is the situation for all nuclei/grains, also the faster growing cube type. Other explanations for their faster growth must therefore be sought.

Deformation and recrystallization of single and bi-crystals

This work is aimed at studying the effect of the orientation of individual grains and grain-grain interactions on deformation and recrystallization. Single crystals and a bi-crystal of high purity aluminium (99.99%) have been deformed by channel die compression at room temperature. The orientations of the samples have been chosen to represent the major texture components found in polycrystalline aluminium cold rolled to reductions greater than 50%, namely the so-called Cu-, S- and Brass- orientations for the single crystals, and a combination of Cu- and S- orientations for the bi-crystal. The structures (both after 80-90% cold rolling with subsequent annealing) are characterized over a wide range of scale by optical microscopy, SEM

(scanning electron microscopy) and TEM (transmission electron microscopy). The local orientations and misorientations are examined using EBSP (electron backscattering pattern) in SEM as well as by micro-diffraction and Kikuchi pattern analysis in TEM.

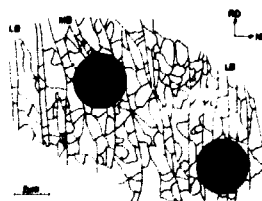
The orientation dependence of the deformation structure observed in the Cu- and S- single crystals agrees well with that observed in the Cu-/S- bi-crystal. The Cu-orientation has been found to develop a heterogeneous deformation structure containing shear bands associated with relatively large local misorientations, whereas the deformation structure of the S- orientation is less heterogeneous and with smaller misorientations. The study of nucleation and growth demonstrates that the shear bands in the Cu-orientation crystal are preferred nucleation sites but that the growth rate of such nuclei varies greatly. As a reflection of the differences in the deformation structure the nucleation and recrystallization in the S- crystal is postponed compared with that of the Cu-crystal. The grain boundary in the Cu-/S- bi-crystal is not a strong nucleation site except where intersected by a shear band.

Metal fatigue

Cyclic slip localization in metal fatigue

One of the major predictions of the static model of persistent slip bands (PSBs) was recently confirmed quantitatively by independent TEM based measurements of a stress-strain proportionality for PSBs at different temperatures. The key assumption in the static model is that the edge dipole clusters, accumulating during cyclic hardening as a result of cross-slip, act as

Sketch showing how, for heavily deformed polycrystals, recrystallization nuclei will be surrounded by matrix material of different orientations. This leads to a broad misorientation distribution in heavily deformed metals.



impenetrable obstacles unless destabilized by internal stresses. The assumption of impenetrable obstacles has been highly controversial for more than a decade. An essential reason for this controversy lies in the widespread belief that penetration is required to explain two very well-documented observations: (1) the extreme homogeneity of slip in the matrix structure embedding the PSBs, and (2) the inverse proportionality between the edge dipole density of PSB walls and the square of the equilibrium wall spacing at different temperatures.

In continued theoretical studies of the dislocation mechanisms of cyclic slip localization, the static model was therefore supplemented with an extended dynamic model. This new model provides a simple quantitative account of observations (1) and (2) without invoking slip penetration or adjustable parameters. The dynamic model is therefore fully consistent with the static model, so the combined static-dynamic model accounts for the above three major observations in cyclic slip localization. Models based on penetrable obstacles have not yet accounted for these observations. The static-dynamic model is now being examined as a possible theory of 'early' cyclic saturation, which extends to a cumulative plastic shear of about 200. Its major prediction in this context is that the matrix continues to undergo irreversible hardening throughout early saturation. Existing models of saturation depict the matrix as being perfectly passive in saturation, with a fixed volume fraction of PSBs carrying the imposed plastic shear amplitude. In complete contrast, the static-dynamic model depicts early saturation, not as a steady-state, but as a process of continued formation of new PSBs in an active matrix, whose shear amplitude e_M decrea-

ses gradually, as strain is transferred into PSBs, during cycling toward a cumulative shear of 200.

Slip localization in copper at intermediate plastic shear amplitudes

(in collaboration with the Cavendish Laboratory, University of Cambridge, UK)

Based on the static-dynamic model, it has become clear that a dislocation theory of cyclic saturation requires models of matrix hardening and PSB nucleation and growth, in addition to the model of the stress-strain behaviour of PSBs. The development of such a theory obviously requires systematic quantitative mechanical and microstructural characterization of cyclic hardening and saturation. As a first step, such a study was completed for [123] single slip oriented copper single crystals, cyclically strained at constant plastic shear amplitude $e_{pa} = 0.003$ and at room temperature. The crystals were deformed to 40, 400, 1100 and 5500 cycles of strain, the cycle numbers were chosen according to information available in the form of fatigue mechanism maps. At 1100 cycles, nucleation of PSBs was observed to occur by collapse of matrix veins into single PSB walls, as expected. More interestingly, observations were frequently also made of a distinctly different mode of nucleation in which veins split into pairs of PSB walls. Although this localization mode is well known, it has previously been reported only for much lower amplitudes e_{pa} . In the present study, the observations of splitting were all made at the intermediate value of $e_{pa} = 0.003$ at the fairly large cumulative plastic shear of 66, corresponding to 5500 cycles of strain, and hence well into early cyclic saturation. Our electron

microscopy clearly confirms the prediction of an active matrix with a slowly decreasing amplitude e_M during early saturation. In addition, the prediction of an active matrix explains various observations reported in the current literature.

Deformation and fracture of composites

Frequency effects on fatigue of CMC's

(in collaboration with the University of Michigan, USA)

Uniaxial tensile tests of a continuous fibre-reinforced ceramic matrix composite (CMC), consisting of continuous Nicalon SiC-fibres in a calcium aluminosilicate matrix, were conducted in the fibre direction at various loading rates. Faster loading resulted in a higher tensile strength. The fibre pull out lengths at the fracture surfaces decreased with increasing loading rate. These effects could be explained by simple analytical micromechanical models: the interfacial frictional shear stress increases significantly with increasing loading rate (i.e. with increasing interfacial velocity). Such a finding has profound implications on our understanding of CMC's.

The fatigue behaviour during uniaxial tension along the fibres has been studied for a wide range of loading frequencies. Increasing loading frequency leads to a significant temperature increase due to friction at the sliding fibre/matrix interfaces. It was found that the fatigue life decreases with increasing loading frequency. This suggests that the fatigue damage is controlled by a temperature or velocity-dependent interfacial wear mechanism, such as abrasive wear along the fibre/matrix interface.

The importance of the interfacial shear stress on the composite fatigue behaviour was demonstrated in a series of experiments where specimens were immersed in oil (prior to cycling) in order to lubricate the fibre/matrix interface. The specimens that had been immersed in oil survived 10^8 load cycles, whereas non-lubricated specimens failed within 10^6 cycles when tested

under identical conditions. This clearly supports the suggestion that the evolution of fatigue damage in CMC's is strongly controlled by interfacial sliding.

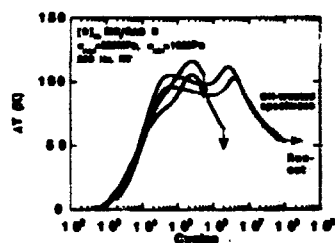
Internal stresses in MMC's

Internal stresses play an important role in the deformation of metal matrix composite materials

(MMC's). A thorough understanding of the generation and possible subsequent relaxation of these stresses is important for the application of today's composites and for the development of even better composites in the future. Much of the Department's current modelling efforts in this field is based on the finite element method (FEM). This is a numerical technique capable of predicting macro-



The fracture surface of a unidirectional CMC displays significant fibre pull-out, showing that the interfacial bonding and friction is low



Temperature rise versus number of loading cycles during fatigue of MMC's. The change in temperature indicates that interfacial shear stress changes during fatigue. G.I.-treated specimens survive 10^7 cycles without failure; untreated specimens fail within 10^6 cycles.

scopic materials' properties as well as providing detailed data on the local stress/strain environment around individual inclusions. These modelling efforts are supported by in-situ neutron diffraction studies of the internal strains.

In 1994, an investigation based on a composite of aluminium reinforced with 5, 10 and 15 vol% SiC whisker has been completed. In-situ neutron diffraction studies revealed that the thermally induced residual strains depend linearly on the volume fraction. This is in good agreement with the results of analytical models. The residual strains in the matrix are in the range of $5-8 \times 10^{-4}$ for the three volume fractions, while the compressive strains in the inclusions are in the range of $10-18 \times 10^{-4}$. In-situ deformation to a total strain of 10^{-2} showed how the elastic straining of the matrix was rapidly saturated, while elastic strain in the inclusions grew during the whole deformation process. FEM calculations based on so-called cell modelling gave

results which correlated well with the experimental data.

It was observed that while the thermally induced residual stresses did have a pronounced effect on the composite phase deformations within the initial $1-2 \times 10^{-2}$ total strain, the effect essentially vanished upon further straining. This closely follows FEM-prediction, neglecting the initial residual stresses.

In-situ neutron diffraction measurements of the elastic phase strains were also conducted upon un-loading subsequent to the 10^{-2} total straining and showed the matrix reversing to a state of residual compressive stresses, while the inclusions reversed to a state of tension. Hence a detailed picture of both thermally induced mismatch strains and deformation induced, residual phase strains in the composite is now available.

In-situ neutron diffraction studies of the relaxation of internal strains in composites has been extended in 1994 to cover the same three volume fractions as above, and in-situ thermal cycling has been performed at 100, 200, 300 and 400 °C. A preliminary evaluation of the results reveals that relaxation of elastic phase strains progresses up till approximately 200 °C, which appears to be a critical temperature for retaining mismatch strains between the ductile matrix and the rigid inclusions.

Modelling residual stresses in layered chromium coatings

The utilization of protective coatings is of key engineering interest. Fracture mechanics studies have recently become of interest for optimizing industrial coating procedures. Surprisingly perhaps, the approaches of continuum mechanics have proven applicable in the failure analysis of

even very thin coatings.

Coatings may fail due to residual stresses originating from the manufacturing process or because of internal stresses produced by thermal or mechanical loading of the component. Chromium coating on a steel substrate is an example of a hard, wear and corrosion resistant surface coating. It is well-known that electroplated chromium exhibits biaxial tensile



Determination of the changes in internal strains during loading/un-loading and thermal cycling of MMC's has been performed using in-situ neutron diffraction in 1994.

stresses in the coating due to a volume contraction in the deposited layers. Technologically interesting applications, such as turbine blades or vanes, involve chromium coatings being applied to thin substrates. However, when thick chromium coatings are applied to thin substrates, contraction in the plated chromium layers causes contraction in the substrate. Thus, during the process, there is a

gradual increase in the rigidity of the substrate. The increasingly larger tensile stresses in the material to be deposited last results in a residual bending moment in the coating, which will ultimately cause the coating to spall off from the substrate.

The residual stress which build up during processing, as well as the details of the fracture processes in through-thickness cracking and interface debonding have been analyzed using continuum mechanics elasticity theory. The model for stress build-up, together with the fracture analyses, provides insight into the sequence of failure events which finally leads to detachment of the coating. The insight provided by the analysis is being extended to allow design and tailoring of thick, crack-free coatings for wear and corrosion resistant brittle coatings on ductile substrates.

Deformation of polymer composites

Polymer composites, based on thermoplastic materials of PPS (poly-phenylene-sulphone) and PES (poly-ether-sulphone) with fibres of glass, are studied under long term creep loading, with the aim of evaluating procedures for design.

The composites contain 21 and 15 vol% short glass fibres in PPS and PES matrices, respectively. The fibres are reasonably well aligned, with some scatter produced during fabrication by injection moulding. The creep testing is performed at temperatures between 20 and 140 °C, at creep stresses of 20 to 120 MPa and in environments of air and water of different pH-values. Most testing is carried out by loading along the fibre direction, supplemented with some off-axis creep testing.

The experimental results are analyzed using four different



Side-view of cracked and spalled multi-layered chromium coating on a thin steel substrate (scale 1:1). The internal stress build-up and the subsequent fracture processes have been analyzed using continuum mechanics elasticity theory.

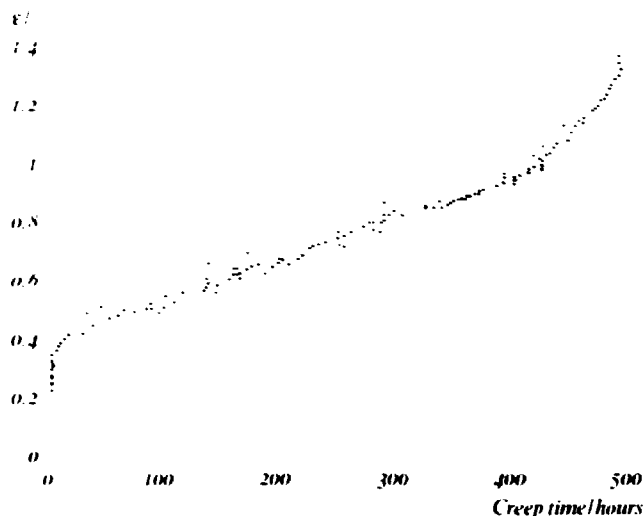
procedures which are related to potential applications for the polymeric composites and to design tasks. The allowables in design are often a maximum strain and/or a lifetime. The individual creep curves of strain as a function of time are analyzed by a mathematical equation including a visco-elastic part, a linear part and an accelerating part for the creep strain. The equation has six parameters. These are analyzed in relation to stress, temperature and environment. In the figure is shown an example of the fitting of the equation to the experimental data, as well as the equation with its numerical parameters. Additional characteristics of the creep curve, such as the minimum creep rate, are derived from the equation. On the basis the creep-equation and the dependence of

its parameters on external conditions (stress, temperature, environment), it is possible to calculate the creep curves for a given set of material parameters and external conditions, as well as to derive additional creep characteristics. To demonstrate the convenience and accuracy of the mathematical relationship, a series of creep curves have been re-calculated and compared to the original experimental creep curves: the agreement is acceptable, with the calculated curve slightly lower than the experimental curve.

In some design tasks the requirement may be a maximum allowable strain and the lifetime to achieve this strain. For pre-selected strains of 0.8, 1.0, 1.2 and 1.4%, the lifetimes are read off the experimental creep curves. These times are also calculated from the mathematical equation, based on the derived parameters. A comparison shows reasonable agreement, and thus allows such lifetimes to be calculated. Global modelling of all creep curves may also be carried out in a continuous iterative process, this being based on the principles of a neural network. The neural network uses a unique method based on weightless look-up tables: the neural network optimizes the model by the principle of cross-validation. The results are again illustrated by comparison with the individual experimental creep curves.

These different procedures are useful for the analysis of a large family of creep curves under a series of external conditions. Each procedure has advantages and disadvantages and it will be the designer's choice to select the method best suited to the actual design task. These analyses may also form the basis for the establishment of norms and standards in creep durability.

$$\epsilon = 0.2965 + 0.107662(1 - e^{-0.165033t}) + 0.00125868t + 7.66188 \cdot 10^{-7}(e^{0.0252251t} - 1)$$



Design of composites for potential load-bearing applications requires analysis of the response to stress, temperature and environment. The figure shows an example of fitting of the six-parameter equation to the experimental data for loading of short glass fibre strengthened PE at 24.5 MPa in water at 90 °C.

Irradiation damage

Defect accumulation during 3 MeV proton irradiations

*(in collaboration with
Forschungszentrum Jülich,
Germany)*

In order to test the validity of the 'Production Bias' model, further irradiation experiments have been carried out. For this purpose, irradiations with 3 MeV protons were chosen since they are expected to produce some multi-displacement cascades as well as single defects in copper. The clusters produced in these small cascades should, according to the production bias model, enhance the accumulation of vacancies (i.e. void swelling) compared with the void swelling observed in the case of 2.5 MeV electron irradiations where all vacancies and self interstitials are produced as single

defects. Furthermore, the accumulated void swelling under 3 MeV proton irradiations should be smaller than that observed under fission neutron irradiations. This is because the clustering propensity under neutron irradiations is likely to be significantly higher than that under 3 MeV proton irradiations. To verify these predictions, specimens of pure copper were irradiated with 3 MeV protons at 523 K to dose levels of about 0.002, 0.008 and 0.01 dpa. Preliminary post-irradiation investigations using transmission electron microscopy and positron annihilation techniques clearly show that the accumulation of vacancies in the form of voids in copper irradiated with 3 MeV protons is higher and lower than that observed, respectively, under 2.5 MeV electron and fission neutron irradiations. These results are taken to be further support for the production bias model.

Effect of radiation damage on hardening and deformation behaviour

*(in collaboration with Harwell
Laboratory, UK)*

Deformation experiments on irra-

diated copper and copper alloys have shown that even a relatively low dose irradiation with fission neutrons at about 320 K causes drastic changes in mechanical properties. The initial yield stress is greatly increased and the ductility is reduced to practically zero due to irradiation. Instead of work hardening, the irradiated materials exhibit work softening and localized deformation. Furthermore, the post-deformation microstructural investigations of the irradiated materials show an almost complete lack of homogeneous dislocation generation during deformation. These results are not consistent with the traditional hardening model based on Orowan type of hardening mechanism. Therefore, a new model is being considered in terms of 'Cascade Induced Source Hardening'. The model considers the trapping of small gliding clusters/loops (produced directly in cascades) at the grown-in dislocations. If these dislocations get decorated by a large number of closely spaced loops during irradiation, then the grown-in dislocations are likely to become locked and may not be able to act as dislocation sources during deformation. Preliminary calculations of the break-away stress necessary to unlock a decorated dislocation suggests that the observed irradiation-induced increase in the initial yield stress can be qualitatively explained within the framework of the present model.

Electroceramics

Electrically conducting ceramics are studied as model and/or candidate materials for solid oxide fuel cells, oxygen sensors and oxygen separation membranes. The prime methods of characterization

are ac- and dc-conductivity measurements, thermo-voltage measurements and structural characterization by X-ray and neutron diffraction.

Ageing (decrease) of the oxygen ion conductivity of yttrium stabilized zirconia (YSZ) was studied continuously over a period of 600 hours; the decrease amounted to 20-30%.

The van der Pauw conductivity measurement geometry was applied to thin plates (100-200 microns) of YSZ, lanthanum-strontium manganites and proton conducting Nafion membranes. A mathematical description of the 4-terminal ac response was obtained for the alternating current case.

Seebeck coefficients (thermo-electric power) of representative oxide ceramics for solid oxide fuel cell application were studied as a function of doping level, temperature and oxygen partial pressure. Transitions from p-type conduction to ionic conduction and from ionic- to n-type conduction in going from oxidizing to reducing atmosphere, were neatly probed by the thermoelectric measurements. Theoretical descriptions of the phenomena were obtained as the final result of a PhD study on the Seebeck effect.

Progress was achieved in understanding the so-called Meyer-Neldel law, which, for many series of related materials reveals an unexpected link between the pre-exponential term and the activation energy for the diffusion- and conduction processes.

Perovskite type oxides were studied with respect to conductivity and structure. A series of rare earth aluminates based on La, Nd, Sm, (NdDy), Gd, Tb, Er, or Y, were produced by the glycine route. Additions of 5% Mg to the aluminates were made to produce substitution on the B-sites; this

ensured creation of a fair amount of oxide vacancies and allowed the influence of minor impurities on the conductivity to be ruled out. All these ceramics had a p-type contribution to the electrical conductivity in air, but at lower oxygen partial pressures they are pure ionic conductors. Correlations between ionic radius of the rare earth element and the conductivity were found. Structural studies of the similar perovskites, $\text{SrCeO}_{3-\delta}$, $\text{SrCe}_{0.85}\text{Y}_{0.15}\text{O}_{3-\delta}$, Mg-doped TbAlO_3 and YAlO_3 , $\text{LaAl}_{0.95}\text{Mg}_{0.05}\text{O}_{3-\delta}$ and $\text{NdAl}_{0.95}\text{Mg}_{0.05}\text{O}_{3-\delta}$, were carried out using a combination of single crystal X-ray, powder X-ray, conventional neutron diffraction and time of flight neutron diffraction. Detailed information on the oxide ion positions was obtained. All these perovskites deviate slightly from cubic structure.

Using high temperature X-ray diffraction, we have studied phase relations and thermal expansion of a number of systems including strontium ferrates, doped and pure samarium oxide, Ca-doped ceria, single crystals of YSZ, and lanthanum chromite ceramics. The in-situ reduction of NiO-YSZ composites into Ni-metal cermet at 1000°C was also studied.

The fundamental studies of structure and conductivity of oxide ceramics are made in part in collaboration with the Centre for Materials Research, Oslo and the Norwegian Institute of Technology, Trondheim as well as the Rutherford-Appleton National Laboratory, UK.

Electron and optical microscopy

Electron microscopy

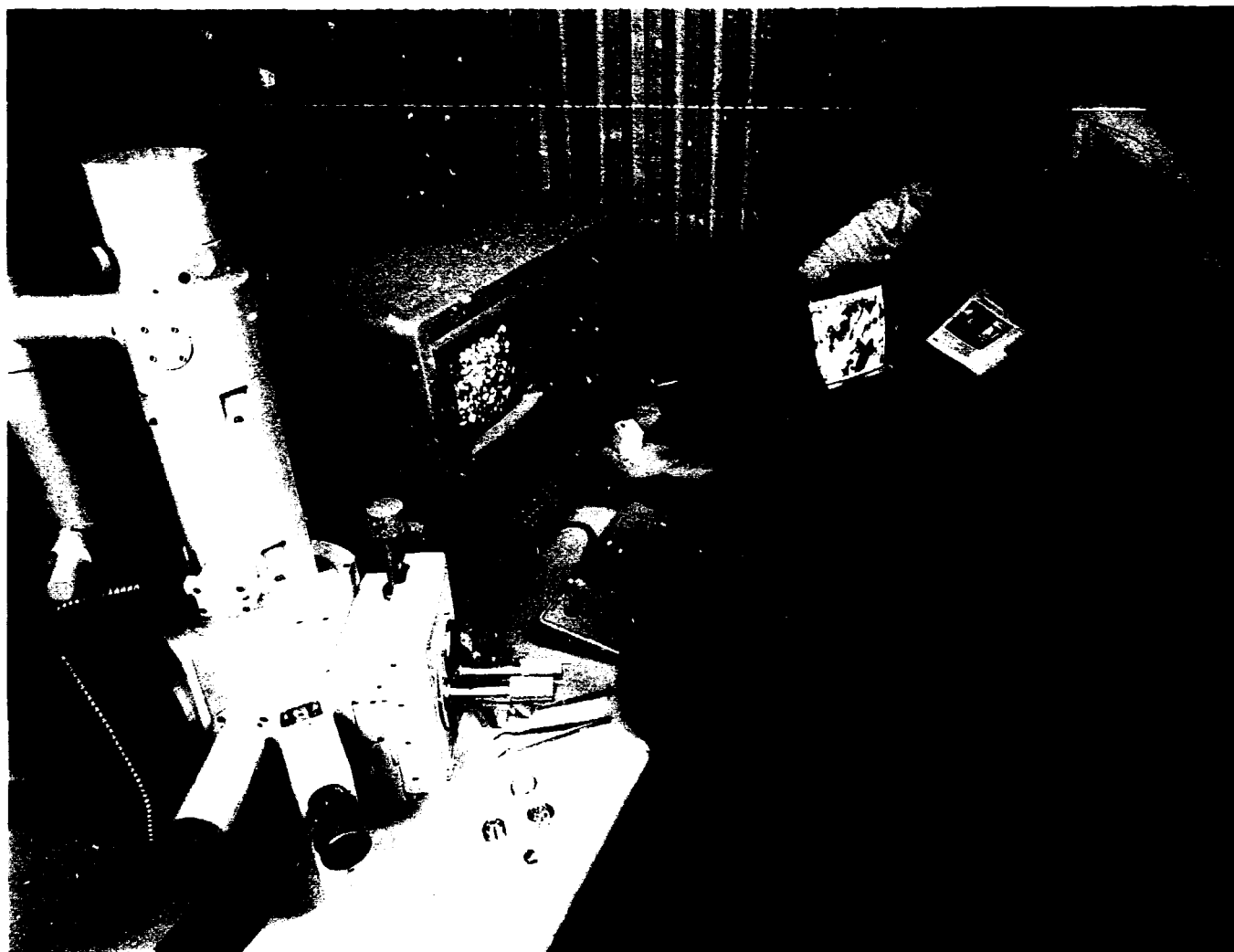
A JEOL 53101V scanning electron microscope was installed in the

Department in December. The microscope can be operated both under high vacuum and under low vacuum in the range up to 2 Torr. In the low vacuum mode, insulators can be examined without it being necessary to apply a conductive coating of carbon or gold. The microscope will primarily be used by the solid oxide fuel cell group for studies of SOFC materials and components of SOFC stacks.

A Gatan slow scan CCD (charge coupled device) camera was installed on the JEOL 2000FX transmission electron microscope. The camera can be used for acquisition of diffraction diagrams as well as low intensity images. Algorithms for automated analysis of diffraction and Kikuchi patterns are being developed.

Equipment for in-situ deformation of specimens in the ElectroScan environmental scanning electron microscope has been developed in collaboration with the Engineering Department, University of Cambridge, UK. The equipment can be used for testing in tension, compression and bending; extension/deflection measurements and signal from the integral load cell allows strain and stress measurements to be related to events observed during the in-situ deformation. A stage for in-situ studies of crack extension in brittle materials and crack face bridging in composites has also been constructed.

A simple and rapid method for use in transmission electron microscopy for determining the orientation of, and misorientation between, grains or sub-grains has been developed. The method has been extended to be used for determination of the normals to planar structures and their traces.



Newly purchased JEOL 5310LV scanning electron microscope (SEM). This low vacuum microscope can operate at chamber pressures of 2 Torr, allowing insulators to be imaged without the application of a conductive surface coating of carbon or gold.

Analytical electron microscopy

Energy Dispersive X-ray Spectrometry (EDS) allows chemical analyses to be carried out in the transmission (TEM), scanning (SEM) and environmental scanning (ESEM) electron microscopes. EDS involves analyzing the characteristic X-ray spectrum which results from interaction of the high energy electrons with the specimen. Since the electron beam may be finely focused, chemical information may be obtained from very small

areas of the specimen.

In SEM and ESEM it is possible to obtain chemical information simultaneously with a characterization of the surface topography and microstructure of the investigated material. As an example, this is often used within the SOFC-project where chemical reactions between the cell components and the pore distribution of the porous electrode materials need to be established because they are some of the factors which ultimately determine the SOFC-cell performance.

When EDS is combined with TEM, chemical information may be obtained from single grains and grain boundaries. This is used for investigations of inhomogeneities in the materials and segregation of elements to the grain boundaries.

Parallel electron energy loss

spectroscopy (PEELS) is another analytical technique which may be associated with TEM. PEELS may be used for chemical analyses and it is especially useful for analysis of light elements such as oxygen. The technique is rather new and PEELS equipment does not yet exist at the Department, or elsewhere in Denmark. However, this powerful technique is used, by members of the Department in collaboration with the Department of Materials Science and Metallurgy at the University of Cambridge, UK. This PEELS work concentrates on a chemical and physical characterization of the transition elements in solid solution in an oxide ion conductor; specifically, Mn in yttria stabilized zirconia. The conductivity of an oxide ion conductor is related to the oxidation state of the transition metal in



solid solution. Since the transition elements may easily change their oxidation level, there are various possibilities in their behaviour in the host lattice. This, and the role of the transition metal in the oxide host is yet to be established.

Optical microscopy and digital image processing

Qualitative improvement of optical microscope images and quantitative image analysis have become possible with the installation of a Zeiss digital image acquisition and database system in 1994. An advanced image processing software package has been purchased to meet the various image processing tasks within the Department.

These facilities have been used extensively, for example to provide quantitative characterization of the microstructure of solid oxide fuel cells (SOFC). Electron microscope and optical microscope images complement each other to a certain extent, because the differences in contrast between the constituents are based on the electron density and the light reflectivity, respectively. For instance, the lanthanum strontium manganite cathode (LSM) of the SOFC cathode can be distinguished from YSZ in the optical microscope while it cannot be distinguished from YSZ in the

A Zeiss digital imaging and database system was purchased in 1994. This equipment is used in the quantitative characterization of, for example, grain size, grain shape, particle- and pore-size and volume fraction of a second phase.

electron microscope image. Apart from being complementary, qualitative and quantitative analysis of optical microscope images is also a cost efficient way of defining region-of-interest sample areas prior to further microstructural and microchemical characterization by electron microscope techniques. Sample preparation is critical for obtaining good results by both imaging techniques and procedures for preparation of difficult samples such as thin and soft SOFC electrodes deposited on very hard and brittle substrates have been established.

Methods and procedures have been developed for determination of grain and void size distribution and variation of the form factor. Empirical relationships are being established for estimating the number of three-phase boundaries in SOFC electrodes. This number may show a correlation to the electrode electrochemical performance.

Non-microscopical characterization techniques

Neutron diffraction studies of texture

The neutron diffraction work was mainly concentrated on texture and internal strain measurements.

During the year, the software for texture presentation was upgraded allowing in-situ plotting of pole-figures and kinetic data. Neutron beam time was used for various texture measurements

such as:

- i) texture gradients through the flange in a tubular component manufactured by radial extrusion. Strong correlations between the position in the formability diagram and the type of texture developed were observed.
- ii) texture in hot deformed aluminum alloys as a function of strain, strain rate and temperature. This was part of a BRITE/EURAM project.
- iii) texture development during grain growth in Cu, brass and Fe. Neutron bulk textures were compared with local orientation measurements obtained by electron back scattering pattern analysis (EBSP) to provide data to help in the development of models for texture dependent grain growth. This was in collaboration with researchers from TU Bergakadem Freiburg, FRG and was supported by the EC Large Installation Programme.

Neutron diffraction studies of internal strains

In 1994 the stress-rig for in-situ neutron diffraction studies of internal stresses in crystalline materials has been up-graded with a new system for thermal loading. The rig is now equipped with two infrared heating elements which are capable of heating test specimens to approximately 400 °C. Hence thermal and mechanical loading can now be applied simultaneously during in-situ experiments.

The design phase for a new

multi-wire neutron detector for the instrument has been completed during 1994, and most components have been produced. The detector will be in operation in early 1995, and is expected to enhance the data acquisition rate by a factor of ~15. This will allow improved time resolution in studies of relaxation and creep, improved measurements of small volume fractions and poor neutron scattering elements as well as reduced beam time costs for commercial investigations.

Apart from applications discussed in section 2.9 and 3.5, a number of internal stress investigations have been conducted within the user programme sponsored by the EU Large Installation Plan.

Synchrotron radiation studies

Synchrotron irradiation experiments provide very intense and highly collimated photons in a continuum from the far infrared to the high energy X-ray region (1 eV- 500 keV). The useful number of photons in diffraction experiments is typically 3 to 6 orders of magnitude higher than the comparable number of neutrons in neutron diffraction studies. The penetration depth can furthermore generally be tuned from 10 Å to 1-10 cm by varying the energy.

These features open up for exciting new possibilities for materials characterization. In consequence, the Materials Department has, during 1994, defined a research programme for synchrotron radiation studies. The programme includes projects related both to fundamental research and materials technology. Investigations will start in 1995, with experiments taking place at the synchrotrons HASYLAB in Hamburg and

ESRF in Grenoble.

One point of focus will be local strain and texture measurements with probe volumes that might ultimately be of the order of micrometers. Such studies would be relevant for determining internal stresses in metal matrix composites and metallic multilayers, for studies of growth kinetics of single grains during recrystallization, and for three-dimensional orientational imaging. Other applications are foreseen within the field of solid oxide fuel cells and characterization of point defect clusters in radiation damaged materials.

Positron annihilation

The positron annihilation technique is a useful experimental method for studies of defects in solids. This is because positrons, injected into a material, may be trapped at vacancy type defects (i.e. vacancies and clusters of vacancies, gas bubbles, dislocations, surfaces, interfaces etc.) and this trapping can be detected by different experimental methods. The measurable characteristics of the annihilation of the positrons give information about the defects in which the trapping took place. Various positron annihilation spectrometers are available in the Department, viz. three lifetime, one Doppler broadening and one angular correlation spectrometer. As part of the development of the experimental techniques, new positron sources for lifetime experiments have been successfully produced in collaboration with the ISOLDE group at CERN by implantation of the positron emitter ^{22}Na into platinum and aluminium foils. In particular the 'platinum source' is stable at much higher temperatures (~700 K) than conventional 'sandwich' type sources, even in the presence of oxygen.

Annealing experiments on helium implanted copper have been continued as part of a collaboration with KIT Karlsruhe, FRG, to study the development of the microstructure and He bubbles in particular. In collaboration with Research Centre Jülich, Germany, proton irradiation of copper at 525 K and subsequent measurements have been carried out in order to compare the defect structures with those obtained in previous electron and neutron irradiations under the same conditions.

As one of the projects under the Centre of Powder Metallurgy studies of ultrafine powders of FeCr have been carried out, in particular on the effects of the reduction of surface oxide on the powder grains. In collaboration with Northwestern University, USA, a study of nano-crystalline palladium and copper has been initiated with the aim of obtaining information about the porosities in these new materials which have been produced by compaction of ultrafine powders.

Studies of free volume at interfaces in polymer blends as well as test measurements to define the reproducibility of positron lifetime spectroscopy in measurements on polymer materials have been started in collaboration with Chalmers University of Technology, Sweden.

The work on ceramic materials was continued with measurements on the annealing behaviour of various low-temperature electron irradiated ceramics in order to investigate materials of different structures and compositions and the effect of electron energy. Furthermore, taking advantage of the newly developed positron sources, equilibrium measurements were carried out to study the influence of oxygen partial pressure.

Materials Engineering – modelling and performance



A thorough knowledge of the mechanical properties of engineering materials is essential for the design of advanced components and structures. Of special interest are polymer composite materials, metal matrix composites and engineering ceramics. The research activities in Materials Engineering are centred around structural mechanics analysis of destructive and non-destructive materials testing procedures. A considerable number of the projects are carried out in close collaboration with Danish and European industrial partners and often directly related to improving the performance of a particular component or of a new material combination.

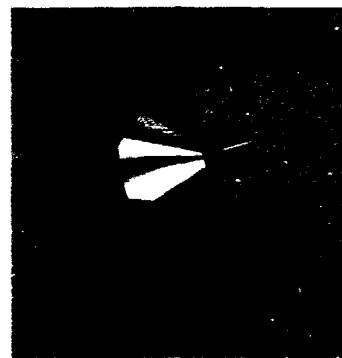
Design and evaluation of composites

Design with polymeric composite materials

A conceptual study for a new type of flywheel for energy storage in vehicles and for stationary purposes has been completed in 1994. The design was financed by the national energy research programme EEP-92. The storage capacity for flywheels is determined by the polar inertial moment and the rotational speed. A characteristic stress for the flywheel is the tangential stress in the rim, and the larger the allowable stress allows higher rotational speed. For a given material with a given allowable stress, it can be shown that the storage capacity is proportional to the volume of the rim. This means that materials

with a high strength to weight ratio also will give the highest energy storage capacity per weight unit. Continuous fibre/polymer matrix materials are therefore obvious candidates for this specific application, and the design is based on high-strength carbon fibres and a thermoplastic matrix.

One of the main objectives of the project has been to design the flywheel as simple as possible. The material for the rim is continuous carbon and glass fibres in a res-matrix and the rim is attached to an integral steel disc and shaft. The rim and disc attachment is through a thin carbon fibre/res shell with a small conical angle or a cylindrical shell. The connections between the shell and the disc and



Flywheel design – potent application pending – for energy storage in vehicles and for stationary purposes. The design is based on high-strength carbon fibres and a thermoplastic matrix. Peripheral speeds of up to 675 and 820 m/s, for vehicle and stationary applications respectively, achieve corresponding energy storage capacities of 0.14 and 0.23 MJ/kg.

rim are made as adhesively bonded joints. The wheel is designed for large gyro moments and centrifugal forces. The design allows for peripheral speeds up to 675 m/s for vehicle purposes and 820 m/s for stationary applications; energy storage capacity is 0.14 MJ/kg for transportation applications and 0.23 MJ/kg for stationary applications.

A patent has been applied for on the details of the design and the design concept has been accepted by an international technology group for automotive applications.

Impact behaviour and damage tolerance for composites

Polymer matrix fibre composite panels, shells and sandwich configurations are normally designed for in-plane loading or for evenly distributed weak surface loading. Such structures are generally considered to be vulnerable with respect to concentrated loading and impact loading. However, impact loading cannot be avoided for general engineering applications and due to the favourable strength-to-weight ratio for these materials, they are candidates for materials for protective purposes against impact and even for ballistic protection, i.e. armour materials. It is therefore important to investigate the behaviour of this type of material under impact loading and to study the influence of this type of incident on the strength and stiffness properties of the material, their damage tolerance.

The damage tolerance of thin laminates and sandwich panels has been investigated within the framework of international BRIT/ECRAM programmes as given below.

In the DISIR-programme, which finished in 1994, both laminates

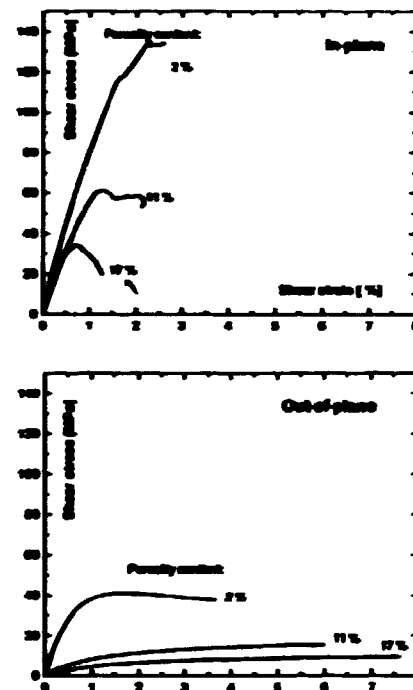
and sandwich have been studied with the aim of the detection and characterization of the damage as well of repair methods.

The DAVITOS-programme (Damage Tolerant Design of FRP Sandwich Structures) aims at establishing prediction methods for fatigue of damaged sandwich components. Both carbon fibre and glass fibre skin layers and honeycombs as well as foam core materials were considered in the total project, with each collaborating partner only carrying out studies on a few material combinations. Each partner manufactured their own specimens. For the tensile or compression fatigue loading, the specimens were 200 mm long and 100 mm wide, and the specimens were damaged by a controlled impact energy using a drop-weight testing stand; subsequent tests were performed in the laboratory. In addition to the mechanical testing, finite-element analysis of the damage development under cyclic loading has been initiated.

The impact behaviour of continuous fibre polymer composite materials has been investigated in the LUCHO-project 'Light Weight Armour Optimization'. The Department has manufactured a large amount of panels with different configurations of glass fibres (unidirectional and fabrics) and different matrix materials; these specimens are for general use in the project by all partners. The panel thickness ranges from 2 mm up to 34 mm, and they are manufactured with different degrees of porosity content (2-18 vol%) which changes the impact behaviour and the total weight of the panels.

Extensive mechanical testing has been performed on the materials used, aiming at establishing data for numerical analysis of the impact phenomena. The start-

Mechanical properties for polymer matrix fibre composite panels with variation in porosity. Normally, only the in-plane properties are considered, but for impact loading the out-of-plane loading behaviour is also important



ing point has been static testing with strain rates of about 10^{-4} s⁻¹ covering both conventional tension and compression testing as well as shear testing. Traditionally, only the properties in the panel's plane, the in-plane-loading are considered, but for impact loading the out-of-plane is also important. To determine the out-of-plane shear stiffness and an estimate of the out-of-plane shear strength, the double notched beam test has been used. Both the shear stiffness and strength is highly sensitive to the porosity content. The reduction is largest for the out-of-plane properties where both stiffness and strength are reduced by a

factor of 10 and 4 respectively. In contrast to this, the strain to failure becomes larger for the higher porosities.

The loading rates during static testing are not representative of the loading rates experienced during impact incidents, and tests with strain rates up to 8 s^{-1} have been performed for one of the material combinations. The stiffness of the tested material does not change within the range tested, but the strength is larger for the high strain rates.

Damage tolerant CMC's

(in collaboration with the University of Michigan, USA)

Damage tolerant ceramic matrix composites (CMC's) have potential for load carrying structures at high temperatures. Most of the technologically interesting applications involve varying loads. It is therefore very important to understand the behaviour of ceramic matrix composites during cyclic loading. Traditionally, fatigue run-out is defined to be 10^6 cycles. There are however examples in the literature that fatigue failures occur after 10^6 cycles. In order to determine whether an endurance fatigue limit exists, it was therefore chosen to perform cyclic tension-tension tests up to 10^8 cycles. It was found that a fatigue limit does indeed exist. All fatigue failures were found to occur within a few million cycles. Specimens that survived 10^7 cycles all survived 10^8 cycles.

The effect of stress ratio ($R = \sigma_{\min} / \sigma_{\max}$) was also investigated. At a given maximum stress a higher minimum stress leads to a longer fatigue life. Also, the fatigue limit (defined as run-out for 10^8 cycles) increases as the minimum stress is increased. This is con-

sistent with the hypothesis that fatigue damage evolution is controlled by an interfacial wear mechanism since the amount of interfacial sliding decreases with decreasing stress range.

Mechanical properties of tooling steels

Tools used for forging and extrusion are typically exposed to very high loads as a consequence of the deformation pressure on the specimens and the often rather complex geometries of the tools. A method to pre-stress the tools or tool inserts is to use so-called 'strip winding'. Here, the tool material is pre-stressed as a radial pressure is successively built up on a ring-shaped core by winding steel bands around the core. Tool steel manufacturers supply mechanical data on the steels as properties measured under bending loads. The uniaxial tensile and compression properties are often unknown, these properties being much more complicated to measure because of the very high strength of the materials. However, in order to predict the performance and the lifetime of the tools it is important to know these properties since the finite element codes and the analytical modelling methods are based on a knowledge of such uniaxial data. A large series of mechanical testing have been carried within the framework of the Danish Materials Technology Programme (MUTP) in collaboration with an industrial partner, Danfoss A/S. Uniaxial tensile and compression tests were carried out in a test machine having high stiffness. Low cycle fatigue tests in combined tension compression were carried out under either strain- or stress-controlled cyclic loading. The results show that by pre-stressing the tools the strain

levels can be closely controlled and thereby forced into ranges where the fatigue life time is considerably improved. The results from the project are now used in the design and modelling work on tool materials used in the industrial production. The modelling and the experimental techniques are being used and improved in a continued collaboration between the Materials Department and Danfoss A/S.

Mechanical properties of fusion materials

Effect of irradiation on mechanical properties and microstructure of molybdenum alloys

(in collaboration with University of London, UK and Pacific Northwest Laboratories, Richland, USA.)

Effects of neutron irradiation on microstructural evolution and mechanical properties of two molybdenum alloys (TZM and Mo-5% Re) have been investigated. All specimens were irradiated with fission neutrons to a dose of $\sim 0.2 \text{ dpa}$ at temperatures between 323 and 723 K.

In both alloys the microstructure was found to be dominated by a high density of irradiation-induced small loops; the loop density decreased rapidly with increase in irradiation temperature. The loop density in Mo-5% Re alloy was higher than that in TZM. In TZM, a moderate density of (111) rafts of loops were observed in Mo-5% Re alloy irradiated under identical conditions.

The Vickers hardness measured at room temperature increased due to irradiation by about 30% and was almost independent of irradiation temperature and cluster density. In general, the Mo-5% Re alloy gave higher hard-

ness values than 17M.

The tensile strength of both alloys was doubled as a result of irradiation. However, both alloys lost their ductility after irradiation to only ~ 0.2 dpa. The loss of ductility is thought to occur due to the weakness and not embrittlement of grain boundaries. It is suggested that the increase in yield strength is due to the difficulty in generating dislocations (source hardening) rather than due to the clusters acting as obstacles to dislocation motion (obstacle hardening).

***Effects of irradiations
on mechanical properties
and microstructure of copper
and copper alloys***

*in collaboration with Pacific
Northwest Laboratory, Richland,
USA*

Copper and copper alloys are currently being considered as candidate materials for the first wall and divertor components of ITER (International Thermonuclear Experimental Reactor). The investigation of the effects of neutron irradiation on microstructure and mechanical properties of these alloys is a part of the ITER research and development programme.

Tensile specimens of pure copper and three copper alloys (i.e. CuCrZr, CuNiBe and Cu-Al₂O₃) were irradiated with fission neutrons in the DR3 reactor at Risø at 320 K to doses between 0.001 and 0.2 dpa. Tensile properties and Vickers hardness of both irradiated and un-irradiated specimens were determined at 295 K. Pre- and post-deformation microstructures were investigated using a transmission electron microscope. The fracture surfaces were examined in a scanning electron

microscope.

The yield strength and Vickers hardness of pure copper and Cu-Al₂O₃ increase very rapidly with irradiation dose and reach saturation already at a dose of 0.1 dpa. In this case of CuCrZr and CuNiBe alloys there is no indication of saturation up to 0.2 dpa. There is, on the other hand, a rapid decrease in ductility with increasing irradiation dose in pure copper as well as copper alloys. Already at a dose level of 0.2 dpa they lose their ability to deform plastically in a homogeneous fashion. A high density of precipitates seems to delay the onset of plastic instability and work softening.

The analysis of mechanical properties and microstructure suggests that the increase in the initial yield stress due to irradiation may arise from the strong pinning of dislocation sources (e.g. grown-in

The determination of the mechanical properties of irradiated materials is an important part of the fusion materials effort. Materials are irradiated in the DR3 reactor or in collaboration with installations abroad. Tensile and fatigue testing is then carried out using specially shielded equipment in the newly built controlled testing area shown in the picture.

dislocations). The loss of ductility appears to be related to the intrinsic weakness of the grain boundaries compared to the hard grain interiors and not to grain boundary embrittlement due to impurity segregation.

In view of the available information on mechanical properties and microstructural data, Cu-Al₂O₃ and CuNiBe appear to be more suitable alloys for application in ITER.



Modelling and design of SOFC stacks

Modelling of SOFC stacks

A mathematical model describing the heat-, mass- and charge-transfer processes in an operating sofc-stack can be a valuable tool in the process of designing, building and operating an sofc-stack. When made in the form of a computer simulation program the model allows studies of changes in stack performance with changes of design or choice of materials. This is far easier than carrying out such changes to the actual sofc-stack. The model can also be of use when interpreting results of experimental tests on real sofc-stacks.

During the last year the model describing the spatial temperature-, current density- and reactant-distribution in an sofc-stack has been further developed to include methane as fuel. This allows treatment of the problems expected for operation of a methane fuelled stack, i.e. large thermal gradients resulting in large thermal stresses expected due to the strongly endothermic nature of the steam reforming process (where methane reacts with water to form carbon monoxide and hydrogen) taking place inside the stack.

The stack model has been used in a number of studies of the importance of various design parameters, and in the planning of suitable operation conditions of the 500 W stack, that is to be assembled during 1995.

When optimizing the materials for use in sofc one of the challenges is the requirement to meet, simultaneously, a number of conflicting demands. The interconnector plates must show good electronic conductivity, gas tightness (good sinterability), chemical stability in both reducing and

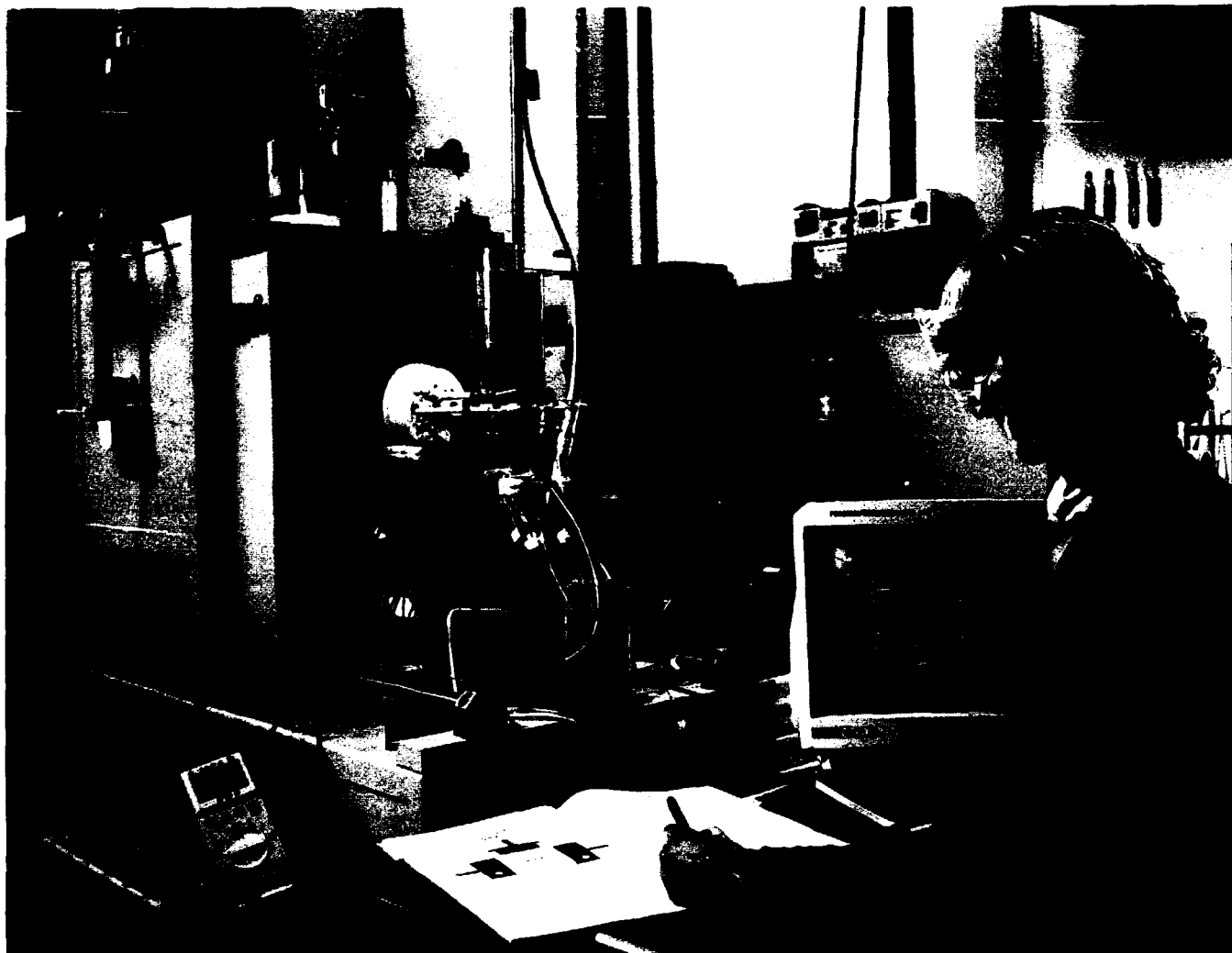
oxidizing environments as well as chemical and mechanical compatibility with the other materials used in the stack. Lanthanum chromite doped with strontium or calcium is able to satisfy these requirements. However, when choosing the degree of doping one faces conflicting requirements. The electronic conductivity as well as the sinterability greatly increases with dopant concentration, as does the extent of matching of thermal expansion coefficients of the chromite and the electrolyte material (yttria stabilized zirconia). This is desirable, as it results in smaller residual stresses when the stack is cooled. However, Ca- or Sr-doped lanthanum chromite expands when exposed to the reducing environment in the anode compartment of an sofc stack; this expansion increases with increased doping and leads to the build up of mechanical stress in the stack during operation.

A simple model describing the build up of stresses in the stack due to mismatches between the thermal expansion coefficients of the materials, and due to the above described dimensional instability of the interconnect material has been set up. Suitable levels of doping, as judged from a purely mechanical point of view, have thereby been identified.

Metallic interconnects for solid oxide fuel cells

A search for a suitable metallic alloy to substitute the presently employed ceramic LaCrO_3 based interconnect material has been undertaken. The purpose of the interconnect in a fuel cell stack is to separate the fuel gas (hydrogen or natural gas) from the air gas while at the same time conducting electrons from one cell to another. The much lower prices of potential

metals, much better heat and electrical conductivities, and better mechanical properties are reasons for the search for a metallic interconnect. However, there are stringent requirements for such a metal: it must have a thermal expansion coefficient close to that of other cell components, it must have very good corrosion resistance, especially against oxidation and carburization, and the interactions with the electrode materials must not be detrimental for the cell properties. These requirements put a severe limit on possible alloys. Amongst a number of high-temperature alloys a Cr-rich alloy, dispersion strengthened by fine Y_2O_3 particles shows the best properties and is now being tested in more detail. Besides the effect of strengthening, the Y_2O_3 also greatly improves oxidation resistance. The thermal expansion coefficient of the Cr-rich alloy is slightly larger than that of the more critical cell component, namely the yttria stabilized zirconia electrolyte, which will be beneficial as this ceramic then will be in a state of compression after the stack assembly. The alloy forms Cr-oxide as a protecting scale. However, even though the Cr-oxide is a moderate electronic conductor, the scale thickness cannot be allowed to exceed more than a few micrometers. Also, the evaporation rate of Cr-oxide is rather high at elevated temperatures and this can destroy the electro-catalytic properties of especially the cathode. Activities are now focused on lowering the stack assembling and operation temperatures as well as performing controlled surface modifications such that the resulting surface oxide has a low evaporation rate and low electrical resistance.



The picture shows the testing of an SOFC stack. The stack is placed in the grey furnace to the left. The wires coming out of the furnace are voltage probes and thermocouples which are used to continuously monitor the stack performance. These signals are displayed by the computer to the right.

Materials selection for SOFC-stacks

A solid oxide fuel cell stack is an all solid state device. The basis of its invention, about 50 years ago, was a solid oxide electrolyte which consists of a mixed oxide (a ceramic) of Y_2O_3 and ZrO_2 , usually called yttria stabilised zirconia (YSZ). This material conducts oxide ions but is a good electronic insulator. The electrical resistance is only low enough at temperatures in the

range of 900-1000 °C and even then the electrolyte film has to be only about 0.2 mm thick.

For construction of a fuel cell stack, at least three more components are needed, namely a positive air electrode, a negative fuel electrode and a cell interconnector containing channels for fuel and air. A number of requirements have to be fulfilled for the materials of these components.

- 1) The thermal expansion coefficients must match reasonably well as the components must be sintered together into solid contact.
- 2) They must all be chemically and dimensionally stable in the range from room temperature to the sintering temperature of about 1300 °C; there must be long term stability at about 1000 °C in air and hydrogen (fuel) in the case of the electrolyte and the inter-

connector, and in air for the cathode, in fuel for the anode.

- 3) The electrode materials must have good electrical conductivity and the ability to convert the fuel and oxygen electrochemically (i.e. be good electro-catalysts) in order to get a low enough internal resistance in the cell. The interconnect must be a good electronic conductor and must be impermeable to ions and gases. These demands have resulted in the following material choices: Anode (fuel electrode): A mixture of Ni-metal and YSZ. Cathode (air electrode): Lanthanum strontium manganese oxide. Interconnect: Lanthanum strontium chromium oxide. This selection is that most usually used in SOFC prototypes internationally. However, work is still going on in order to find even better materials and materials

combinations. For example, certain chromium based metal alloys are not far from meeting the requirements for the interconnect (see previous section).

Materials for incineration of biomass and waste

When incinerating biomass and waste in power producing plants, high temperature corrosion is often observed in key components like superheater tubes. The fuel contains compounds of chlorides, sulphates and alkali metals which cause corrosion processes which are detrimental at temperatures higher than 450°C.

In a Danish project on superheater materials for waste incineration using straw and other vegetation, the corrosion of selected materials is studied by exposing probes in operating incineration plants. After 600 hours of exposure the materials loss is determined to be in the range of 0.1-2.5 mm/1000 h using optical microscopy. These large differences in material loss are partly an effect of material composition and environment in the plant. It is observed that low alloyed boiler steels have less corrosion resistance compared with higher alloyed steels, the latter having corrosion loss approximately 2-3 times lower. The changing flue gas environment in the different plants is a cause of some of the large differences in metal loss observed. However, the relative performance of the tested materials is similar from plant to plant.

Observations in the environmental SEM and conventional SEM are made on the base material, the corrosion layers and the deposits from the exposure tests. The deposits and corrosion layers investigated show heavy attack of chlorine and sulphur-rich com-

pounds, which are known to be very corrosive. In the corrosion layers, iron is absent due to evaporation of iron-chlorides, while nickel and chromium remain as different compounds of chlorine, sulphur and potassium. More conclusive information on the effect of the individual elements will be available at the end of the project.

For further documentation on which materials perform best a test superheater has been constructed for scaled-up, long time exposure of candidate materials. This test superheater is constructed of 4 different materials, where each will be exposed at 4 different temperatures in the range 420-540°C. It will be possible to evaluate the materials performance found in the small scale tests. At the same time, data on corrosion resistance will be produced which could be used in the design of both new incineration plants and the renovation of old plants.

Non-destructive testing methods

Ultrasonic scanning

The ultrasonic scanning system HFUS 2000 has been improved during 1994. A digitizer module together with completely new scan-software make new types of scanning possible. Earlier, it was only possible to measure peak echoes in pre-selected gates but now the whole echo pattern can be captured and presented as A-scan (one shot) or B-scan (multiple A-scans from a line). These techniques are especially useful for testing carbon/epoxy laminates for delaminations and are typically used as a supplement to C-scan.

The scanning system has been used for the inspection of many different materials including a

large number of glass fibre/foam sandwich specimens as part of a BRITE/EURAM project. The sandwich plates have high ultrasonic damping which makes the traditional pulse echo technique impossible. Instead a through-transmission technique was applied using separate pulser and receiver transducers. This technique is sensitive to defects such as impact damage resulting in debond between the skin layer and the foam core.

X-ray radiographic non-destructive testing (NDT)

The performance of many new structural materials depends on microstructural perfection. Even minor flaws such as cracks, voids, inclusions and other inhomogeneities may cause catastrophic failure on loading. Traditional engineering materials such as steel are also subjected to increasing wear and corrosion rates and there is therefore a strong motivation for the development of NDT techniques which can characterize the development of defects with time.

For these reasons, the activities within the field of radiographic NDT have increased considerably in 1994. The activities have concentrated on the development of techniques for making improved use of radiographic image information. This involves very different tasks such as the determination of ceramic sample density differences and of residual wall thickness in steel pipes. Image processing is of central importance and algorithms and software have been developed for calibration and retrieval of quantitative data from radiographs. X-ray radiography is also important for NDT of light-element composites such as carbon-fibre reinforced ceramic matrix composites. High contrast

and high spatial resolution is obtained combining low energy radiography on fine-grained X-ray film with digitization and image processing. For instance, crack development within fracture toughness tested samples may be quantified. Low energy radiography is also suitable for detection of slight compositional differences in rubber.

SDI is also important for quality assurance of advanced ceramics. A central activity has been to predict ceramic sample bend strength based on data on the defect population as detected by microfocus X-ray radiography and surface analysis. The potential for using SDI data to predict sample strength has been demonstrated, and future activities will aim at integrating the SDI activities in many materials testing procedures. The quantitative SDI data will be used for process optimization. Prediction of residual lifetimes will become possible by incorporating finite element modelling, mechanical testing and microstructural analysis.

Determination of residual stresses in components

In collaboration with Danfoss A/S an experiment has been conducted to verify the capabilities of both X-ray and neutron diffraction techniques to determine engineering stresses in structural components. A simple curved beam sample was subjected to a known macroscopic deformation and subsequently investigated by both X-rays and neutrons. The neutron diffraction measurements revealed how this technique is capable of monitoring the complete stress/strain profile through a structural component in close agreement with a well-known analytical solution, while the X-ray tech-

nique showed its capability to monitor surface stresses, also in good agreement with the analytical solution.

In collaboration with Alfa-Laval Separation a series of neutron diffraction measurements were conducted on a commercial centrifuge component. The residual strain state in a region



close to a mounting hole was investigated by neutron diffraction before and after a test loading, which is commonly applied prior to shipment of the centrifuge. The non-destructive neutron diffraction measurements revealed the presence of residual strains in the as-produced centrifuge part, and a large intensification of these strains due to the test loading. Results are now being compared to FEM-modelling results in order to improve the design of the component.

Apart from these investigations, both related to the MUR-programme on 'Modelling of the Mechanical Behaviour of Materials and the Practical Application of the Models', commercial investigations of residual stresses in structural components have also been conducted for foreign companies during 1994.

The determination of residual stresses in structural components by neutron diffraction. Incident and diffracted beam cross-sections are defined by apertures made from cadmium. The apertures are positioned close to the sample surface to enhance the spatial resolution. The experimental configuration is here exemplified by a set-up used for the investigation of residual stresses in a welded steel component.

Materials Technology – synthesis, processing and products



The manufacture of advanced materials components often requires new processing, fabrication and joining techniques. Pilot plant studies of the production of fibre reinforced polymer composites, fine-powder metallurgical components and thin ceramic layered structures demand the construction of specialized equipment. This research and development also provides test specimens of new advanced materials for other programmes of the Materials Department. The research activities in Materials Technology involve the manufacture of components of polymer matrix composites, engineering ceramics, prototype solid oxide fuel cells and fine-powder metals. Brazing and bonding techniques are being applied to a variety of these materials. Many of the research programmes are carried out in collaboration with other Danish and European research organizations and industrial partners.

Manufacturing processes for advanced composites

Manufacture of polymer composites

In the manufacture of continuous fibre reinforced polymer composites, the involvement of the Materials Department serves mainly three purposes: a) the study of the fundamental principles of filament winding, autoclave processing, and resin transfer moulding, b) fabrication of test specimens, and c) development of prototype components. The processing equipment consists of a

computer controlled filament winding machine, a hot-air-high-temperature pressure autoclave, and equipment for resin transfer moulding.

Two projects on manufacturing technology for thermoplastic composites with continuous fibres have been completed and reported: One project on filament winding and another project on fibre preforms (fibre preforms are pre-shaped structures which have not yet been consolidated to the final component). Fibre preforms can be woven, knitted, braided, or stitched together.

In the filament winding project, a new hot chamber filament winding technology has been developed and investigated. Basically, the technique consists of winding the materials (fibre and matrix) onto a mandrel at the process temperature. The incoming tape is preheated with hot air, and by applying a tensile stress to the tape, the consolidation is enhanced. The mandrel and the already wound material are kept at the process temperature until the entire specimen has been wound. Hot chamber filament winding has been investigated on carbon fibre/PEEK (poly-ether-ether-ketone) and on carbon fibre/PP (polypropylene). After optimization of the process there is perfect consolidation, a porosity content of ~2 vol% and an interlaminar shear strength of 18 MPa. High material quality is also obtained in 10 mm thick tube specimens wound at a speed of 8 m/min.

In the preform project a new hybrid yarn of glass fibres and thermoplastic polyester fibres has

been developed in collaboration with v/s Kaj Neekelmann, Silkeborg, Denmark. The hybrid yarn is a co-mingled textured yarn consisting of structural fibres and thermoplastic polymer fibres. In a subsequent heating and consolidation process the plastic fibres melt and become the matrix material in the formed fibre composite material. The hybrid yarn is a so called postpreg material (opposite prepreg) since the fibres are impregnated with the matrix in a post-process. The hybrid yarn is 'soft' and drapeable, and therefore very suitable for producing fibre preforms by almost any textile technology.

In the current project the hybrid yarn is a mixture of glass fibres and polyester fibres. It is essential, for ease of production, that the hybrid yarn is a thorough and homogenous mixture (at the fibre level) of the two types of fibres. The flow distance of the molten matrix is then small, which means that the time at temperature may be short, and/or the consolidation pressure may be small. The glass fibres and the polyester fibres have been co-mingled by an air jet texturing technology. Fabrics are woven from the hybrid yarn, and laminates are fabricated by autoclave consolidation at different pressures. Microscopy, measurements of fibre content, porosity and mechanical properties show that the quality of the material is very high in all the laminates and that there is potential for a very wide industrial use for structural fibre composite components. As a case study, a car door panel has been fabricated by a 'cold' pressing technique of pre-heated fabric woven from the hybrid yarn. This study was done in collaboration with KIEWIT Industri, Hadsund, Denmark, and Komposit Procesteknik, Spentrup, Denmark. The material quality

and the results obtained with the hybrid yarn are very promising, and a new three-year project has been started.

In another project on filament winding, an integrated computer tool, CADPATH, which automates the process of designing, analyzing, and manufacturing of filament wound structures has been developed in collaboration with Alfa-Laval Separation A/S, Soborg, Denmark. For geodesic winding on surfaces of revolution, CADPATH calculates winding pattern, fibre path and local wall thickness for a selected fibre angle. Strength analysis can be performed using classical laminate theory or finite element analysis. Finally, CADPATH can calculate the control data

necessary for manufacturing the structure on an SC winding machine. CADPATH has been tested by designing and manufacturing an industrial component: A 'bowl' for a separator centrifuge. The 'bowl', which measures $D176 \times L183$ mm, has been wound on a specially designed dismountable

Carbon fibre/epoxy, filament wound bowl (foreground) for a separator centrifuge (at rear) developed in collaboration with Alfa-Laval Separation A/S. An integrated computer tool, CADPATH, was developed to determine the design, analysis and manufacture of the component. CADPATH calculates and optimizes the geodesic winding pattern, fibre path and local wall thickness.





Damaged armour test specimen. Side view of the plate after projectile impact testing carried out by a project partner.

steel mandrel. A small number of carbon fibre/epoxy 'bowls' have been fabricated and tested in the centrifuge equipment. The carbon fibre/epoxy 'bowl' weighs 345 g (the original aluminium 'bowl' weighed 821 g), and the composite bowl has a much improved safety factor against fracture at 7500 rpm, which is the nominal rotational speed of the centrifuge.

Fabrication of test plates for the EUCLID project on Light Weight Armour Optimization has continued throughout the year. Laminates of glass fibre/polyester, glass fibre/PP, and aramid fibre/PP are fabricated by a film stacking technique. The technique consists simply of stacking layers of woven fibre fabrics and thermoplastic

films of the matrix materials, followed by consolidating the stack into a laminate in a subsequent autoclave process.

Within a BRITE/EURAM II project on Damage Tolerant Design of FRP Sandwich Structures (DAMTOS) a series of sandwich test specimens of glass fibre epoxy skin layers on a 15 mm thick foam core have been fabricated by an autoclave cure process.

Hot working of metal matrix composites

The involvement of the Materials Department in the BRITE/EURAM project 'Forging of Aluminium Metal Matrix Composites (Al-MMCs) for Automotive Components' serves mainly three purposes: 1) development of an understanding of the hot deformation characteristics of the selected

commercial Al-MMC materials, 2) study of the microstructure evolution and the internal stress during hot deformation, and 3) provision of the required information for modelling the forging process and microstructure as well as for optimizing the forging conditions. The materials studied are supplied by Duralcan, and contain 10 vol% Al_2O_3 , 20 vol% Al_2O_3 and 20 vol% SiC particulates respectively. Compression and tension tests have been carried out in the temperature range 20-500°C, and at different strain rates in the range 10^{-3} to $1\ s^{-1}$.

The microstructure of the specimens deformed at room temperature has been examined by SEM. The work on hot deformed specimens is in progress. The evolution of damage of reinforcement particles with increasing strain has been analyzed. The relationship between the level of particle damage and the reduction in stiffness of the material has been investigated. It has been found that: 1) the strain localization and the particle damage localization are affected slightly by the volume fraction but significantly by the strength of the matrix, being more severe in the matrix with higher strength, 2) the mechanism by which particle failure occurs is also greatly affected by the strength of the matrix. For a soft matrix, interface debonding dominates, while for a hard matrix, particle cracking dominates, and 3) the relationship between stiffness reduction R_f ($R_f = 1 - E/E_0$, where E_0 is the initial stiffness) and DF (number fraction of cracked particles) is approximately linear with a slope close to one.

Fabrication of test plates for the EUCLID project on Light Weight Armour Optimization.



Scanning electron micrograph of solidification microstructure of gas-atomized Fe4C containing a small amount of phosphorous. Magnification $\times 1000$.



The changes in microstructure through the forging process have been identified and the effect of the forging process on mechanical properties has been evaluated. It has been found that: 1) the changes in microstructure occurring through the forging process correlate with the history of macro-scale deformation of the material predicted by a FEM model, 2) in general, the forging process reduces the microstructural inhomogeneities such as particle clusters and particle-free bands which results in increased elastic modulus and increased fracture elongation, and 3) further improvement in fracture elongation may be achieved by using raw materials with higher extrusion ratios and by optimizing the post heat treatment.

In-situ synthesis of ceramic composites

Al₂O₃ matrix SiC composite powders have been obtained by an in-situ reaction process using Al, SiO₂ and C as starting materials. Experiments were carried out in the temperature range of 600–1500 °C in an argon atmosphere. Differential thermal analysis and X-ray diffraction results showed that the reaction initiated with the melting of Al and was immediately followed by oxidation. Si was found to be an intermediate reaction product at temperatures lower than 1200 °C, which suggests that SiC formed via a direct reaction between Si and C, after SiO₂ was reduced, not via the SiO phase in this low temperature range. When the temperature was above 1400 °C, mullite was formed. The general reaction finished at 1500 °C. In this temperature range, SiC was likely to be formed through the reaction of mullite and carbon first forming a gas phase SiO and CO, and by a subsequent reaction of SiO and C.

Powder metallurgy

Atomization

A variety of alloys and pure metals have been produced in powder form by atomization. In total, 24 atomization experiments were performed during 1994. Techniques for production of alloys containing reactive components or components with widely separated melting points have been established. Sample extracts are Cu-Zr, Fe-C, Al-Cu-P and Mo-containing steel alloys.

Solidification of the CuSn6 alloy was modelled by an analytical and a numerical method. To verify the models, primary and secondary dendrite arm spacings were calculated as a function of local solidification time and the results were compared with experimental observations on the atomized particles. The modelling fits very well with the experimental results which show a square root and a cube root relation, respectively, for the primary and secondary spacings. As a result of these calculations it was concluded that in modelling of secondary dendrite arm spacings, ripening processes have to be taken into account. The modelling also showed that primary dendrite and

cell spacings are determined by diffusion and solute rejection in the melt, whereas the growth velocity determines the shape of the cells and dendrites and whether the secondary arms are stable or not.

Ultrafine powders

(in collaboration with the Institute of Physics, Technical University of Denmark)

Ultrafine metallic nickel was produced by the evaporation-condensation technique and subsequently passivated by controlled exposure to the atmosphere. The kinetic and thermodynamic properties of this material during heating in inert gas was studied by micro-gravimetry, differential scanning calorimetry and differential thermal analysis. The properties were found to be significantly different from the properties of commercial nickel oxide in that there is a reduced stability and a higher dissociation rate of the ultrafine material. The dissociation upon heating in He was found to be composed of two steps and the activation energies of the steps were measured to be 136 and 26 kJ/mole respectively.

Joining of advanced materials

Brazing of stainless steels and superalloys

The BRIT/EURAM project on the development of brazed novel plate heat exchangers was continued this year together with partners from Denmark, Sweden and Germany. The specially developed corrosion test specimen was used to detect, analyze and measure the corrosion attack from various media on the diffusion zones and the individual phases in a multi-phase brazed joint.

Using the specimen specially designed to simulate a single crosspoint in the plate heat exchanger, the tensile strength of a brazed single crosspoint was measured successfully for a variety of combinations of base metal, brazing filler metals and brazing parameters.

Brazing powder metallurgical materials

After the work on joining powder metallurgical (P/M) parts with commercially available filler metals was finished as planned in the Danish Centre for Powder Metallurgy, a new project was started on the development of new brazing filler metals. The work in the Centre had shown that although a few commercial filler metals could be used, they all had very limited gap filling properties. In addition, they all showed

Optical micrograph of iron based powder metallurgical parts which have been brazed using a newly developed filler metal. The gap between the parts (vertical line) has been properly filled without significant intrusion of the filler metal into the pores. Magnification $\times 75$



drawbacks of either leaving a powdered crust, where the powder filler metal had been placed and/or showed a deep infiltration into the pores of the P/M base material, perpendicular to the gap. The aim of the new project was to develop filler metals which would react with the iron based P/M materials and solidify isothermally to close the pores open to the surfaces forming the gap, thereafter the gap is progressively filled with the filler metal. Using this brazing concept, P/M parts with a theoretical density of only 80% 0.1 mm wide gaps with an area of $35 \times 35 \text{ mm}^2$ have been filled from one side, leaving a nice fillet at the opposite end of the gap. The infiltration of the P/M parts perpendicular to the brazed gap showed to be less than a millimetre.

Joining of ceramics

In the Danish Centre for Advanced Technical Ceramics, the joining programme was finished with the planned investigations of the bending strength of brazed joints in hot pressed silicon nitride. The influence on the four-point bending strength from the brazing parameters, Brazing temperature, gap width and preparation of the surfaces was investigated using a AgCuTi active filler metal in a high vacuum furnace. Median bending strength of up to 465 MPa and a Weibull modulus of 7.1 was obtained.

Contract work

Contract work was continued on industrial applications of dip-brazing of aluminium alloys as well as vacuum brazing of stainless steels, superalloys, ceramics and ceramics to metals. The

growing interest shown by Danish industry for joining ceramics-to-metals has resulted in major development activities using the active soldering and brazing filler metals as well as an active high temperature filler metal.

The great industrial success with a component containing an active brazed ceramic/metal joint has now after the comprehensive development work and testing programmes resulted in major contracts of pre-production of these components, while the high vacuum brazing process has been successfully scaled up and transferred to the industrial company itself.

Manufacture of LEU fuel elements

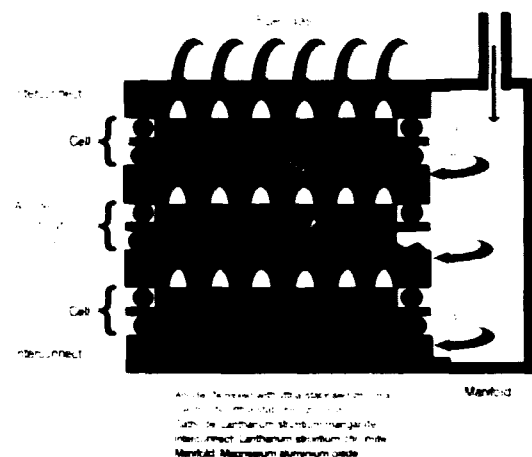
The fabrication of low enriched uranium (LEU) fuel elements was continued in 1994; more than 260 LEU elements have now been fabricated in the Materials Department. In total, approximately 175 LEU elements have been irradiated in the DR3 reactor since the changeover from the use of high enriched uranium (HEU) elements, which started in 1988. All of the LEU fuel elements have performed successfully.

The ultrasonic scanning method for measuring cladding thicknesses has been further improved, and the results have now been presented to the research and test reactors community.

SOFC technology

Manufacture of SOFC components

According to the current stack concept of the Danish solid oxide fuel cell programme, DK-SOFC, a number of different, mainly ceramic, components are required to build an SOFC stack:



Bipolar flat plate design as used in the Danish SOFC programme, exemplified by a 3-cell stack. The reaction in the SOFC is: reduction of air at the cathode ($\text{O}_2 + 4\text{e}^- \rightarrow 2\text{O}^{2-}$), transport of the oxygen ions through the electrolyte to the anode where the fuel is oxidized ($2\text{H}_2 + 2\text{O}^{2-} \rightarrow 2\text{H}_2\text{O} + 4\text{e}^-$). To prevent air and fuel gas from mixing it is necessary to seal the cathode on the fuel gas inlet and outlet side, and the anode on the air inlet and outlet side. It is also necessary to seal the manifolds (only the air manifold is shown in the figure).

- Single cells
- Interconnect plates
- Ceramic glues
- Glass or glass-ceramic seal components and
- Manifolds for fuel-gas and air

Manufacturing techniques have been selected with emphasis on cost and the possibilities for scale up. Complex ceramic compounds are made using techniques which allow continuous production of high quality materials: single cells are manufactured by a combination of tape casting and spray painting techniques; interconnect plates are made by biaxial powder pressing and subsequent machining of sintered components with

diamond tools, ceramic glues are applied by either spray painting or by tape casting and manifolds are made by slurry casting.

The size of single cells and of interconnect plates is presently being scaled up from 45×45 mm to 80×80 mm. This increases the active cell area fourfold and enables building of a stack with a projected power output of 0.5 kW in 1995. Transfer of manufacturing technologies developed at Riso to the Danish industrial partner of the project, Haldor Topsøe A/S, has commenced.

Processing of electroceramic powders

Processing includes both the preparation of the powders and the fabrication of actual components.

The electroceramic items are solid bodies obtained by sintering (welding together the original powder particles into a solid), or by packing the powder particles into a porous body. The solid bodies are used because of their mechanical and electrical properties (e.g. piezoelectrics, capacitors, ionic conductors, electronic conductors for use in extreme conditions), while the porous bodies are used because of their large surface areas (e.g. electrodes, electrocatalysts). Most of the conventional ceramic processing methods can be carried out at the Department and are used for electroceramic powder processing.

The ceramic processing must change shapes and surface characteristics of the powder particles and render them transformable into the final items. Purity and particle shape and particle surface morphology are the main characteristics to be controlled in the starting powders. The X-ray diffraction (XRD) spectra of the

powder while being processed and the electrical properties of the items obtained are the main control parameters of the process. As an example we can cite the processing of a mixture of oxides and nitrates to convert them into electroceramic powders with perovskite structure. The powders are shaped into chromite plates with the typical chemical composition of $\text{La}_{0.5}\text{Sr}_{0.5}\text{Cr}_{0.9}\text{V}_{0.1}\text{O}_3$. The plates are electronic conductors with electrical properties similar to those of graphite and SiC but they have the added advantage of being stable in air and in pure hydrogen at 1000 °C. At 1000 °C graphite burns in air and SiC is decomposed in hydrogen. The plates are a main component in the Solid Oxide Fuel Cells.

The fabrication process developed at Riso includes the dissolution of La, Cr, Sr and V oxides and nitrates into an aqueous solution with the organic complexing agent glycine, followed by conversion by high temperature pyrolysis. The pyrolysis takes place at approx 600 °C in rotary ovens of pilot plant size. The powders produced are not yet electroceramics: the XRD patterns indicate that the perovskite structures have not yet appeared. Heating treatments at 800 to 1200 °C causes the crystals of perovskite to form and to reach dimensions large enough for XRD detection. Scanning electron microscopy and surface area measurements allow a further check of the powder characteristics obtained.

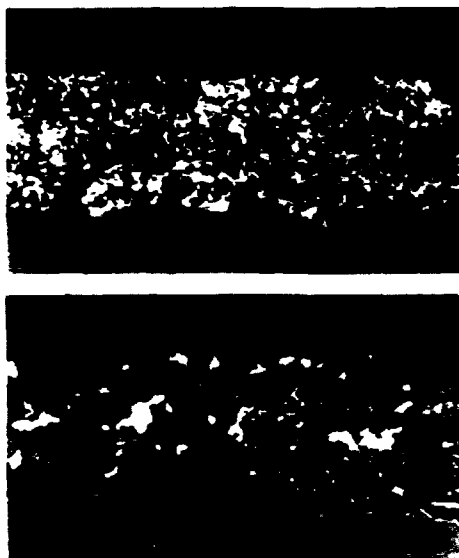
Forming into the shapes of the final electroceramic items can be done by dry pressing into the shape of simple plates 10×10 cm² or by mixing with organic binders and lubricants to mould into more complex shapes. Forming is followed by careful heating to sinter to dense large tough plates that are machined with diamond tools.

Sealing of SOFC stacks

The bipolar flat plate design with external manifolding, as used in the Danish SOFC programme, requires seals to prevent mixing of fuel gas with air.

Glass or glass containing materials are preferred as sealing materials. However, a suitable sealing material must meet a number of requirements with respect to physical and chemical properties. The seal development has, during the course of 1994, been concentrated on systems having phosphate and silicate as glass-formers. The systematic optimization of glass properties as described in the annual report for 1993 led to a number of stable seals showing good performance during real life tests in stacks of 3 to 5 cells.

The study of the influence of the seal on anode performance has received much attention in 1994. Two types of reactions, distinguished in terms of the range of interaction, have been studied. For short range interactions, interface reactions where the seal is in physical contact with the anode, the experiments comprised heat treatment of powdered mechanical mixtures of anode components and sealing materials, followed by determination of possible reaction products by X-ray diffraction. For long range interactions, reactions caused by transport of volatile species from the seal, experiments were performed by heat treatment of anodes placed close to, but not in direct contact with, seal materials. Analyses of anode performance were carried out by impedance spectroscopy both before and after heat treatment. Changes in anode microstructures and chemical compositions were analyzed by scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS).



The experiments showed that seal materials, with phosphate as the only glass former, react with nickel to form nickel phosphide. The reaction is not limited to the anode-seal interface, but occurs over an extended area. The reaction is a consequence of vapour transport of phosphor. Experiments with silica based seals showed no signs of interface reaction and only a weak degradation of the anode performance was suggested by preliminary electrochemical measurements.

Fabrication of high T_c superconductors

Industrial interest in superconducting materials is highly associated with material parameters such as critical temperature, T_c , maximum

Optical micrographs of SOFC anodes cross section, magnification $\times 500$. The upper micrograph shows the normal anode structure with vitreous stabilized zirconia (grey), metallic nickel (white) and porosity (black). The lower micrograph shows that all of the nickel has reacted with the phosphorous to form small islands of nickel phosphide (white).

current density, J_c , and magnetic field strength inside the superconducting phase, H_c . The discovery of $YBa_2Cu_3O_{6-x}$ (YBCO), $Bi_2Sr_2CaCu_2O_{8-x}$ (BSCCO 2212) and $Bi_2Sr_2Ca_2Cu_3O_{10-x}$ (BSCCO 2223) enabled critical temperatures to be realized at liquid nitrogen temperatures (boiling point -196°C). As attainable values for J_c and H_c were found sufficiently high, the materials have become technologi-

cally attractive. Extensive materials development, however, is required before fabrication of superconducting components with optimal properties can be established. Superconducting wires in lengths greater than 1000 meters are presently made in Japan and USSR with critical current densities around 20000 A/cm^2 , but the potential is much higher. SKI Research Centre A/S has made short wires with critical current densities up to 70000 A/cm^2 .

A one year research programme was established in 1994 with the following participants: SKI Research Centre A/S, Risø Materials and Physics Departments, Institute for Mineral Industry at the Technical University of Denmark and the Research Association of the Danish Electric Utilities (DREF). The aim of this programme is to provide a strategic analysis of the potential of high- T_c superconductors in the Danish electric power distribution system and to expand the efforts in the development of national technology know-how. Support for the programme came from the Danish government's Energy Research Programme, the two groups of electricity utilities HIKRAFT and HESAM and from the collaborating participants.

At the Materials Department considerable know-how has been established in the field of complex-based ceramic synthesis through participation in solid oxide fuel cell programmes as described in previous sections. The contribution to the present programme was accordingly focused on the development of precursor powders with reduced crystallite size in order to improve the manufacture of superconducting components. Powders already produced have demonstrated a reduction in size while superconductivity has been preserved.

Finances 1994

U. C. Lind, B. S. P. 1994



The activities of the Department are supported by a combination of direct government funding, focused project funds from national, EU and international programmes and fully commercial industrial contracts.

The numbers given in the tables are in units of 1000 Danish kroner. The equivalent amount in US dollars is also shown (DKK 1000 equals USD 171; alternatively, 1 USD equals 5.85 DKK). Inflation from 1993 to 1994 was small (~2.1%). The Danish kroner was strengthened with respect to the US dollar (~15%) over this period.

In 1994, basic funding to the Materials Department was decreased by 8%. There was also an overall slight reduction in project funding (~3%). The decrease in project funding could easily have been more serious; the Danish technology programme, the 'MUP1' programme in which the Materials Department had been very active, was replaced by a much smaller MUP2 programme with significantly different objectives. The changes resulted in a loss of income of approximately DKK 6 million compared to 1993. Fortunately, this decrease

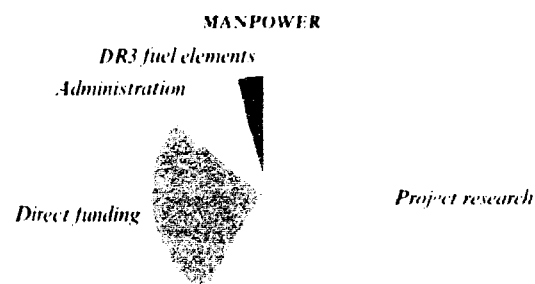
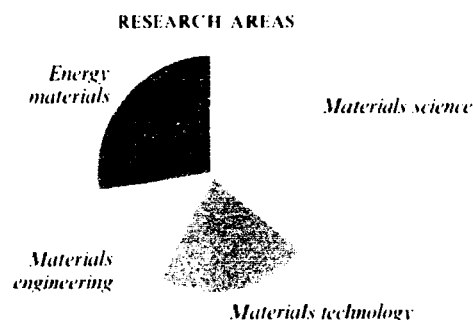
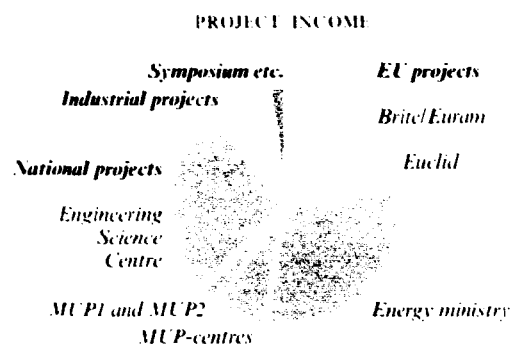
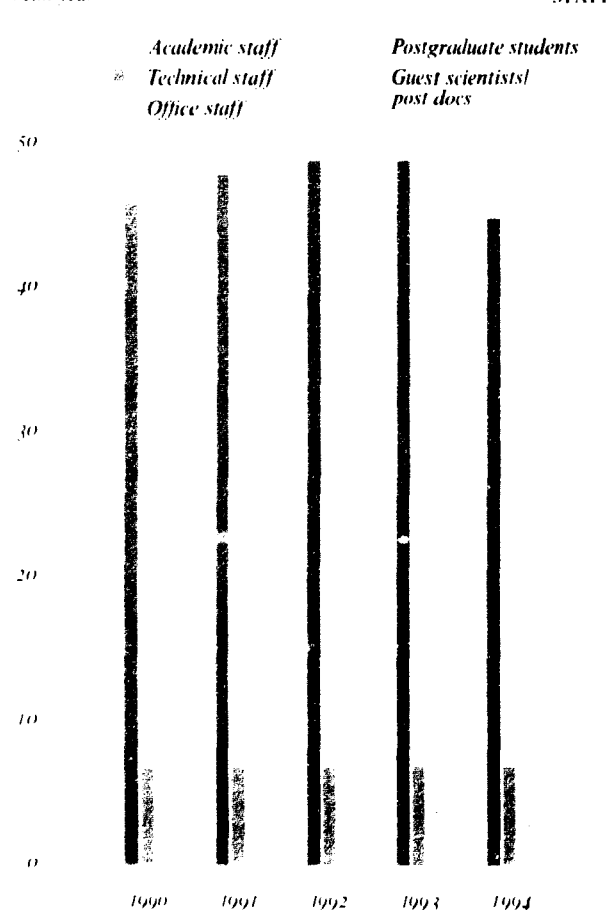
INCOME 1994	DKK 1000	USD 1000
<i>Direct funding (Ministry of Research and Technology)</i>	16 520	2 824
<i>Project funding</i>	38 346	6 555
	54 866	9 379
 EXPENDITURE	 DKK 1000	 USD 1000
<i>Salaries</i>	30 712	5 250
<i>Operating expenses</i>	14 967	2 558
<i>Equipment</i>	5 327	910
<i>External administrative charge</i>	5 224	893
 <i>Total</i>	 56 230	 9 612

was partly compensated for by a general increase in other project areas: the total income to the Department in 1994 was 5% lower than in 1993.

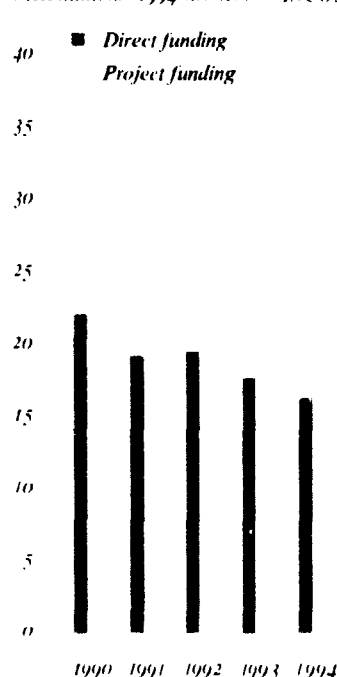
Altogether, 70% of the total income to the Materials Department now comes from sources other than direct grant from the Ministry of Research and Technology. The major project funded programmes are Solid Oxide Fuel Cells (SOFC) and the Engineering Science Centre, which together account for more than half of the total project income.

The policy of controlled growth in the number of tenured academic staff has been continued in 1994, with a net increase of one scientist during the course of the year. The closing down of the Riso hot cell facility in 1994 has meant a reduction of 4 in the total number of technical staff. Educational activities have continued to increase significantly: there were an additional 7 PhD students and 5 more post docs/guest scientists than in the previous year, bringing the totals to 15 and 19 respectively.

Man years



DKK millions (1994-kroner) INCOME



Staff 1994

N. H. Rasmussen



In 1994, 32 staff members left the Department and 22 new members joined () the Department*

Head of Department

Niels Hansen

Scientific staff

Adolph, Eivind
Andersen, Svend Ib
Appel, Charlotte C.
Bagger, Carsten
Bentzen, Janet J.
Bilde-Sørensen, Jørgen B.
Borring, Jan
Borum, Kaj K.
Brøndsted, Povl
Carlsen, Hans
Christensen, Jørgen
Debel, Christian P.
Eldrup, Morten
Gormsen, Steffen
Gotthjælp, Klaus
Gundtoft, Hans Erik
Hendriksen, Peter V.
Horsewell, Andy
Johansen, Bjørn S.
Juhl, Mette *
Juhl Jensen, Dorte
Kindl, Bruno
Kjølby, Sif
Knudsen, Per
Larsen, Peter Halvor
Leffers, Torben
Lilholt, Hans
Linderøth, Søren

Liu, Yi-Lin
Lorentzen, Torben
Lystrup, Aage S.
Løgstrup Andersen, Tom
Mogensen, Mogens
Pedersen, Allan Schrøder
Pedersen, Ole Bøcker
Poulsen, Finn Willy
Poulsen, Jørgen *
Primdahl, Søren
Rasmussen, Karen W. *until 1 June*
Rheinländer, Jørgen *
Singh, Bachu N.
Sørensen, Ole Toft
Toft, Palle
Toftegaard, Helmuth L.
Østergård, Maria J.L. *until 28 Feb.*

Postgraduate students

Ahlgren, Erik *until 15 Dec.*
Bjerregård, Henrik
Carstensen, Jesper Vejlo *
Clausen, Bjørn *
Christoffersen, Henrik
Jacobsen, Torben K. *
Mullit, Paw M. *
Nielsen, Christian Bergenstoft *
Olsen, Henrik *
Poulsen, Jes R.
Pryds, Nini H. *
Ranløv, Jens *until 31 Dec.*
Rasmussen, Torben V. *
Thorsen, Peter A. *
Tiedje, Niels *until 31 Oct.*

Post docs

Godfrey, Andy
Hansen, Ulrich *
Johannesson, Birgir *until 31 Oct.*
Jørgensen, Ole *
Krieger Lassen, Niels C.
Richter, Helmuth
Sørensen, Bent F.
Sørensen, Niels Jakob
Tai, Lone-Wen * *until 30 Nov.*
Teng, Harry Y. *until 4 May*
Winther, Grethe *

Guest scientists

Alcock, Jeffrey R. *until 30 June*
Bonanos, Nicholas
Cai, Shidong *until 3 Aug.*
Carter, J. David *until 15 Sep.*
Dirnfeld, Shraga F. *until 24 Aug.*
Edwards, Danny J. * *until 30 Sep.*
Lebensohn, R. * *until 3 Nov.*
Liu, Qing
Li, Wen-Yu *until 31 Dec.*
Shkerin, Sergey * *until 31 May*
Vulfson, Yuriy *until 30 June*

Consultants

Domanus, Josef *until 1 Nov.*
Nilsson, Tage M.
Waagepetersen, Gaston

Technical staff

Adrian, Frank
Andersen, Axel B. *until 31 Aug.*
Arnsberg, Carsten *until 30 Sep.*
Aukdal, Jørgen A.
Bindner, Jørgen *until 1 May*
Borchsenius, Jens F.S.
Christensen, Carl J. *until 30 June*
Christensen, Peter M. *until 31 Aug.*
Christensen, Svend E. *until 1 Nov.*
Dreves Nielsen, Poul
Eschricht, Lars *until 31 Dec.*
Frederiksen, Henning
Gravesen, Niels Nørregaard
Hansen, Jørgen Lang
Hansen, Niels Jørgen *until 7 Oct.*
Hersbøll, Bent
Jensen, Finn E.
Jensen, Knud
Jensen, Palle V.
Jespersen, John
Kjær, Anne-Mette Heie
Kjoller, John
Klitholm, Cliver
Larsen, Arne *until 30 Sep.*
Larsen, Bent
Larsen, Birgit N.
Larsen, Jan
Larsen, Kjeld J.C.
Lindbo, Jørgen
Mikkelsen, Claus
Nielsen, Birgitte
Nielsen, Ove
Nielsen, Palle H.
Nilsson, Helmer
Nørregaard, Jesper *until 1 May*
Olesen, Preben B.
Olsen, Benny F.
Olsen, Henning

Olsen, Ole
Olsson, Jens O.
Paulsen, Henrik
Pedersen, Børge E. *until 31 Oct.*
Pedersen, Knud E.
Pedersen, Niels Jørgen
Robl, Steen
Sandsted, Kjeld
Strauss, Torben R.
Sørensen, Erling
Aagesen, Sven

Office staff

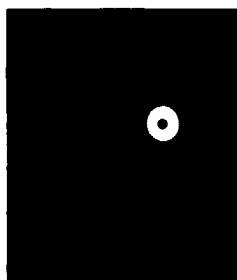
Dreves Nielsen, Elsa
Hoffmann Nielsen, Lis
Larsen, Lisbeth *
Lauritsen, Grethe Wengel
Mortensen, Jytte
Sindholt, Birgitte
Sørensen, Eva M.
Thomsen, Ann

Apprentices

Andersen, Morten
Forné, Anders *until 14 Mar.*
Foss, Heidi E.
Hammer, Christian
Nielsen, Jens Cordt *until 31 Dec.*
Ploug, Martin
Schmidt, Jesper *
Wolfe, Thomas *until 14 Aug.*
Ørgaard, Henrik

Department activities

Paul Vogel, 1997



Postgraduate (PhD) projects

PhD students are affiliated with a university with supervisors both at the university and in the Materials Department. The universities as well as the Riso supervisor are mentioned below.

PhD projects finished during 1994

Franz Bodker
'Magnetic Properties of Nanoparticles'. The Technical University of Denmark, Lyngby.
Supervisor: *Søren Linderoth*

Erik O. Ahlgren
'Thermoelectric Power of Solid Oxide Fuel Cell Materials'. The Technical University of Denmark, Lyngby.
Supervisor: *Finn W. Poulsen*

Niels Christian Krieger Lassen
'Automated Determination of Crystal Orientations from Electron Backscattering Patterns'. The Technical University of Denmark, Lyngby.
Supervisor: *Dorte Juul Jensen*

Jens Rønlov
'Perovskite-type Metal Oxides, Electrical Conductivity and Structure'. The Technical University of Denmark, Lyngby.
Supervisors: *Mogens Mogensen* and *Finn W. Poulsen*

Niels Tiedje
'Formation of Microstructures in Gas-Atomized Cu-6%Sn and 316L Powders'. The Technical University of Denmark, Lyngby.
Supervisor: *Allan Schrøder Pedersen*

Ongoing PhD projects

Henrik Bjerregaard
'Flexible Forging of Metal Matrix Composites'.
This study focuses on hot deformation of metal matrix composites and the correlation between material parameters and process parameters and includes modeling of deformation processes. Also, the microstructural parameters of the materials following forming operations are described.
The Technical University of Denmark, Lyngby.
Supervisor: *Hans Lilholt*

Jesper Vejlo Carstensen
'Structure Development and Mechanisms in Fatigue of Polycrystalline Brass'.
The influence of stacking fault energy on fatigue is studied with polycrystalline brass as a model system. Specimens of Cu-15%Zn and Cu-30%Zn with well-characterized grain size distribution and texture are fatigued at constant plastic strain amplitude. Dislocation microstructures and surface damage evolution are characterized by TEM, SEM and light microscopy.
The Technical University of Denmark, Lyngby.
Supervisor: *Ole Bøcker Pedersen*

Henrik Christoffersen
'Development of Microstructure in Copper during Plastic Deformation'.
The project aims at a detailed description of the microstructural development in deformed copper and its relation to the micromechanics of the deformation processes, for instance the relation to the local variations in strain and slip patterns. It is emphasized that the microstructural characterization is, as far as it is possible, carried out quantitatively.
The University of Copenhagen.
Supervisor: *Torben Leffers*

Born Clausen

'Texture and Anisotropy Effects on the Determination of Lattice Deformation Using Diffraction'

This work focuses on the characterization of polycrystal deformation through modelling and experimental determination of crystallographic deformation of constituents of the polycrystal. The goal is to correlate the microstructural parameters, such as crystallographic structure, anisotropy and texture with the macroscopic characteristics, such as stiffness and flow-stress.

The Technical University of Denmark, Lyngby.

Supervisor: *Torben Lorentzen*

Torben K. Jacobsen

'The Influence of Holes and Notches on the Fatigue Properties of Two-Dimensional Ceramic Matrix Composites with Long Fibres'

The project involves theoretical modelling and experimental measurements of damage around geometric stress raisers in 2-D SiC/SiC and SiC/C woven materials. The modelling is based on the ideas of damage classification and on the models to predict stresses and damage growth using FEM calculations. The models are adjusted and verified by experimental work and observations under fatigue loadings using of acoustic emission, thermovision, and X-ray monitoring.

The Technical University of Denmark, Lyngby.

Supervisor: *Poul Brøndsted*

Jan Berthrendt Ibsen

'Fatigue Life of Off-shore Steel Structures under Stochastic Loadings'

Fatigue crack initiation and crack growth mechanisms in welded joints are studied. A theoretical model to predict fatigue lifetime based on crack closure and residual stress considerations is set up. The model is tested and verified using results from experimental measurements on welded plate specimens and tubular joints.

The Technical University of Denmark, Lyngby.

Supervisor: *Poul Brøndsted*

Paw Møller

'Quantification of the Pore Structure in Porous Materials'

The mechanical and physical behaviour of porous materials such as cement, concrete and brick, is strongly influenced by the shape, size and amount of pores in the material. Although understood qualitatively, quantitative treatments of the relationship between pore stereology and properties need to be developed.

The Technical University of Denmark, Lyngby.

Supervisor: *Andy Horswell*

Christian Bergenstorf Nielsen

'Microstructural Investigation of Electrolytically Deposited Compositionally Modulated Alloys'

The microstructure of copper-nickel multilayers is investigated by transmission electron microscopy. Quantification of grain and grain boundary structures in as-received, heat treated and deformed microstructures will be used to understand the basis for enhanced mechanical properties of multilayers.

The Technical University of Denmark, Lyngby.

Supervisor: *Andy Horswell*

Henrik Olsen

'Control of Ceramic Component Properties through Powder Particle Morphology'

The project aims at achieving a better understanding of the powder particle interaction as a function of powder morphology and sintering conditions. The main objective is to develop a model which can be used to predict porosity, pore size and pore distribution.

The Technical University of Denmark, Lyngby.

Supervisors: *Carsten Bagger and Mogens Mogensen*

Michael Stanley Pedersen

'Superferromagnetic Nanostructures'

This study focuses on the magnetic properties of magnetically interacting nanoparticles leading to a superferromagnetic behaviour of otherwise paramagnetically relaxing particles. The magnetic particles are maghemite, magnetite or amorphous Fe-C ferrofluid. Magnetic Fe-Hg suspended in mercury is also studied. The studies are primarily performed by Mössbauer spectroscopy and magnetization measurements.

The Technical University of Denmark, Lyngby.

Supervisor: *Søren Linderoth*

Chris Pickup

'Internal Stress in Layered Structures'

Copper-Nickel multilayers are being analyzed using neutron and X-ray diffraction as well as convergent beam electron diffraction to determine internal stresses. In addition to improving the techniques for internal stress analysis, the results are to be interpreted in relation to the microstructure of multilayered CuNi.

University of Cambridge, UK.

Supervisor: *Andy Horswell*

Jes R. Poulsen

'Investigation of Defects in Ceramic Materials using Positron Annihilation Techniques'

The main aim of the project is to apply the defect sensitive technique of positron annihilation to obtain information about non-equilibrium defects introduced into ceramic materials by irradiation and equilibrium defects produced at high temperatures under different oxygen partial pressures.

University of Copenhagen.

Supervisors: *Janet J. Bentzen, Morten Eldrup and Andy Horswell*

Vim H. Pryds

'Rapid Solidification of Stainless Steel'. The purpose of these investigations is to characterize the microstructural variations which occur during rapid solidification of stainless steel prepared by melt-spinning and atomization processes. The project also strives to incorporate the microstructural changes observed as a function of cooling rates through a model based on the heat flow balance. The Technical University of Denmark, Lyngby.
Supervisor: *Allan Schroder Pedersen*

Peter A. Thorsen

'The Influence of Grain Boundary Structure on Diffusional Creep'. The structure of a given grain boundary determines its efficiency for absorbing or emitting vacancies. Diffusional creep is therefore influenced by the nature of the grain boundaries present. The efficiency of various grain boundaries in material deformed in diffusional creep is compared with their structure determined by electron microscopical techniques. The University of Copenhagen.
Supervisor: *Jorgen B. Bilde-Sorensen*

Torben V. Rasmussen

'Time Dependent Interface Parameters in Concrete-Based Composite Materials'. This study focuses on experimental techniques and micromechanical models to characterize the bond between a fibre and the surrounding cement-based matrix for a series of fibre/matrix systems. The bond is characterized by a number of parameters. These material parameters will be examined and estimated as a function of time. The Technical University of Denmark, Lyngby.
Supervisor: *Poul Brondsted*

Undergraduate projects

Peter Bloch

'Preparation of High- T_c Superconducting Wires and their Characterization by Means of X-ray Diffraction and Scanning Electron Microscopy'. University of Copenhagen.
Supervisor: *Torben Lethers*

Chris van den Bos

'Software Development for Texture Presentations'. University of Cambridge, UK.
Supervisor: *Dorte Juul Jensen*

Jean-Michel Charmet

'Wear Properties and Grain Size of Alumina and Zirconia Toughened Alumina'. Ecole Nationale Supérieure de Céramiques Industrielles, Limoges, France.
Supervisor: *Ole Toft Sorensen*

Andrew Roger Lee

'Microstructure Characterization of Aluminium Matrix Composites after Deformation'. Brunel University, Uxbridge, UK.
Supervisor: *Yi-Lin Liu*

Henrik Olsen

'Production and Shaping of sinter Cathode Materials'. The Technical University of Denmark, Lyngby.
Supervisor: *Carsten Bagger*

Rozlyn J. Phillips

'Conductivity Study of Perovskite Solid Electrolytes'. University of Waikato, Hamilton, New Zealand.
Supervisor: *Nicholas Bonanos*

Torben Rasmussen

'Atomic Description of Dislocations in Metals'. The Technical University of Denmark, Lyngby.
Supervisor: *Ole Bockor Pedersen*

Tanya Sabin

'Development of SEM Calibration Techniques'. University of Cambridge, UK.
Supervisor: *Andy Horsewell*

Nils Thorup

'The Cathode Process in Solid Oxide Fuel Cells'. Odense University, Denmark.
Supervisor: *Mogens Mogensen*

External lecturers and examiners

Many of the staff members of the Materials Department are actively involved in education and training in materials science as university external lecturers and examiners.

External lecture courses

Jorgen B. Bilde-Sorensen

'Processing Technology, Technical Ceramics', Materials Technology Course, Riso.

Niels Hansen

'Materials Science', The Danish Academy of Engineering, Lyngby.

Jorgen Rheinlander

'Processing Technology, Technical Ceramics', Materials Technology Course, Riso.

Allan Schroder Pedersen

'Processing Technology, Technical Ceramics', Materials Technology Course, Riso.

Ole Toft Sorensen

'Solid State Chemistry', The Technical University of Denmark, Lyngby.

External examiners

Member of the officially appointed corps of Danish university examiners (MSc and BSc):

Svend Ib Andersen

Jorgen B. Bilde-Sorensen

Christian P. Debel

Morten Eldrup

Hans Lilholt

Ole Toft Sorensen

PhD examiner, University of Aalborg.
Svend Ib Andersen

PhD examiner, University of Aarhus.
Morten Eldrup

PhD examiner, The Technical University of Denmark, Lyngby.

Niels Hansen

Hans Lilholt

PhD examiner, Université de Lille, Villeneuve d'Ascq, France.
Ole Bockor Pedersen

PhD examiner, The University of Madras, Madras, India.
Morten Eldrup

Guest scientists and foreign assignments

Staff members on assignment abroad during 1994

Charlotte C. Appel
Department of Materials Science and Metallurgy, University of Cambridge, UK. 15 August - 26 August.

Henrik Bierregaard
Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA, USA. 9 July - 31 December.

Henrik Christoffersen
Department of Materials Science and Metallurgy, University of Cambridge, UK. 3 October - 22 December.

Einn W. Poulsen
Norwegian Institute of Technology, Trondheim, Norway. 1 January - 30 June.

Torben V. Rasmussen
Department of Civil and Environmental Engineering, The University of Michigan, Ann Arbor, MI, USA. 1 February - 15 May.

Bachu N. Singh
Department of Quantum Engineering and Systems Science, University of Tokyo, Japan. 13 - 16 February and 21 - 24 February.

Bachu N. Singh
Research Institute for Applied Mechanics, Kyushu University, Japan. 16 - 21 February.

Bachu N. Singh
Materials Science Division, Pacific Northwest Laboratories, Richland, WA, USA. 25 June - 2 July.

Peder Skov-Hansen
Department of Materials and Metallurgical Engineering, Queen's University, Kingston, Canada. 29 June - 29 August.

Bent F. Sørensen
Department of Mechanical Engineering and Applied Mechanics, The University of Michigan, Ann Arbor, MI, USA. 1 January - 1 May.

Guest scientists at the Materials Department during 1994

Dr. Janis Andersons
Institute of Polymer Mechanics, Riga, Latvia. 5 January - 11 February.

Dr. Claire Barlow
Department of Engineering, University of Cambridge, UK. 3-7 January and 29 August - 16 September.

Dr. Shidong Cui
Department of Applied Physics, Xidian University, Xi'an, China. 1 January - 4 August.

Dr. Sergei V. Divinskii
Institute of Metal Physics, Ukraine Academy of Science, Kiev, Ukraine. 5 - 16 September.

Dr. John H. Evans
Physics Department, University of London, UK. 3 - 15 March and 8 - 19 August.

Mike E. Fitzpatrick
Department of Materials Science and Metallurgy, University of Cambridge, UK. 21 February - 6 March.

Dr. Alan J.E. Foreman
Radiation Damage Department, Harwell Laboratory, UK. 20 August - 3 September.

Dr. Stanislav I. Golubov
Department of Nuclear Materials, Institute of Physics and Power Engineering, Obninsk, Russia. 10 - 17 April.

Clare Hacker
Materials Department, University of Bath, UK. 21 - 29 June.

Alan Heaver
Engineering Department, University of Cambridge, UK. 13 - 22 September.

Peter Holtappel
KFA Jülich, Germany. 15 August - 28 October.

Dr. Darcy A. Hughes
Sandia National Laboratory, Livermore, CA, USA. 4 - 29 July.

Prof. Kevin Kendall
Department of Chemistry, Keele University, UK. 14 - 21 January.

Prof. Peter Klimanek
Institut für Metalkunde, TU Bergakademie Freiberg, Germany. 26 - 30 November.

Dr. Anatolijs Kuzjuevics
Institute of Inorganic Chemistry, Latvian Academy of Science, Riga, Latvia. 1 October - 17 December.

Dr. Ricardo Lebensohn
Instituto de Física, Universidad Nacional de Rosario, Argentina. 1 September - 30 November.

Professor Andreas Mortensen
Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA, USA. 8 August - 2 September.

Dr. Henning Friis Poulsen
HASYLAB DESY, Hamburg, Germany. 1 - 31 September.

Dr. Jean L. Raphanel
LPM-UM-CNRS, Université Paris-Nord, Villetaneuse, France. 10 - 14 October.

Dr. Valerie Randle
Materials Engineering, Swansea University, UK. 18 April - 13 May and 20 - 22 June.

Dr. Sergey Shkerin
High Temperature Electrochemistry, Russian Academy of Science, Ekaterinburg, Russia. 9 March - 9 June.

Prof. Minoru Taya
Department of Mechanical Engineering, University of Washington, Seattle, WA, USA. 31 August - 9 September.

Marie C. Theyssier
Centre CMS, Ecole des Mines de Saint-Etienne, France. 8 May - 20 June.

Susanne Vogel
Institut für Metalkunde, TU Bergakademie Freiberg, Germany. 26 November - 16 December.

Ulrike Wegst
Engineering Department, University of
Cambridge, UK, 13 - 23 September.

Prof. John A. Wert
Department of Materials Science and
Engineering, University of Virginia,
Charlottesville, USA, 5 June - 7 July.

Henri M. A. Wmand
Department of Materials Science and
Metallurgy, University of Cambridge,
UK, 8 - 21 August.

Dr. Philip J. Withers
Department of Materials Science and
Metallurgy, University of Cambridge,
UK, 20 June - 28 August.

Dr. Chung H. Woo
Whiteshell Laboratories, AECL Research,
Pinawa, Canada,
28 August - 9 September.

Camilla Wästlund
Department of Polymer Technology,
Chalmers University of Technology,
Gothenburg, Sweden,
28 February - 11 March.

Dr. Ulrich Zink
The Technical University of Clausthal,
Germany, 28 February - 6 March.

Dr. Steven J. Zinkle
Metals and Ceramics Division, Oak
Ridge National Laboratory, USA,
28 September - 7 October.

Colloquia

'Computer aided investigation of grain
growth', 6 January
Dr. Helmut Richter
Materials Department, Risø National
Laboratory, Roskilde, Denmark

'Properties and crystal structure of
LaCrO₃ based materials', 7 January
Dr. Natsuko Sakai
National Institute of Materials and
Chemical Research, Tsukuba, Japan

'Effects of reinforcement orientation on
tensile response of metal matrix
composites', 7 February
Dr. Niels Jakob Sørensen
Materials Department, Risø National
Laboratory, Roskilde, Denmark

'A static-dynamic model of cyclic slip
localization in low cycle fatigue',
14 March
Dr. Ole Bøcker Pedersen
Materials Department, Risø National
Laboratory, Roskilde, Denmark

'Compositional (functional) gradient
materials and diamondlike nanocom-
posite coatings', 22 March
Dr. Renée G. Ford
Editor-in-chief 'Materials Technology'

'Mechanical properties of layered
structures', 11 April
Dr. Andy Horswell
Materials Department, Risø National
Laboratory, Roskilde, Denmark

'On the structure of fluorite-like oxygen-
conducting solid oxide surfaces and
elements of the energy-band structure of
the system 0.9 ZrO₂ - 0.1 Y₂O₃ (solid
solution)/Pt', 12 April
Professor Sergey Shkerin
Institute of High Temperature Solid
Electrolyte Electrochemistry, Russian
Academy of Science, Eckatherinburg,
Russia

'Research in the field of materials science
in Estonia', 21 April
Professor Priit Kihu
Department of Materials Science and
Engineering, Tallinn Technical
University, Estonia

'The crystallography of grain surfaces in
polycrystals', 26 April
Dr. Valerie Randle
Department of Materials Engineering,
University College of Swansea,
Swansea, UK

'Tension fatigue of continuous-fibre rein-
forced ceramics', 6 June
Dr. Bent E. Sørensen
Materials Department, Risø National
Laboratory, Roskilde, Denmark

'Processing of aluminium alloys for
superplastic forming', 10 June
Professor John A. Wert
Department Materials Science and
Engineering, University of Virginia, USA

'Representing and using boundary miso-
rientation data', 15 June
Professor John A. Wert
Department Materials Science and
Engineering, University of Virginia, USA

'Cluster-assembled nanophase materials',
17 June
Professor Richard W. Siegel
Materials Science Division, Argonne
National Laboratory, USA

'How to combine the worst character-
istics of metal and ceramics', 27 June
Professor John A. Wert
Department Materials Science and
Engineering, University of Virginia, USA

'Time-dependent interface parameters in
fibre-reinforced cement', 25 July
Torben V. Rasmussen
Materials Department, Risø National
Laboratory, Roskilde, Denmark

'Steady state solidification of reinforced
metals', 15 August
Professor Andreas Mortensen
Department of Materials Science and
Engineering, Massachusetts Institute of
technology, Cambridge, USA

'Mechanics modelling of multi-fracture
processes in ceramic composites', 17
August
Professor Y. Jack Weitsman
Oak Ridge National Laboratory and the
University of Tennessee, USA

'Analysis of a particle metal matrix composite subjected to combined thermal cycling and creep loading', 2. September
Professor Minoru Taya
University of Washington, Seattle, USA

'Modelling of extrusion of 600 series Al alloys', 14. September
Dr. Hugh R. Shercliff
Engineering Department, University of Cambridge, UK

'Prospects for synchrotron research at the Materials Department, Riso', 15. September
Dr. Henning F. Poulsen
Hasylab at DESY, Hamburg, Germany

'Synchrotron radiation at Hasylab', 21. September
Professor Jochen R. Schneider
Hasylab, Hamburg, Germany

'Finite element simulation of large elasto-viscoplastic deformation in aggregates with a small number of grains', 11. October
Dr. Jean Raphael
CNRS, Institute Galilee, Université Paris Nord, France

'Modelling low temperature plastic deformation and texture development of Zr-alloys', 18. October
Dr. Ricardo Lebensohn
Instituto de Física Rosario (Rosario Physics Institute), Rosario, Argentina

'Composite mixed conductor membranes for producing oxygen', 16. November
Dr. R. Srinivasan
Air Products, USA

'Substructure analysis in structurally inhomogeneous materials by X-ray and neutron diffraction', 29. November
Professor Peter Klimanek
Freiburg University of Mining and Technology, Institute of Physical Metallurgy, Freiburg, Germany

Social activities

The Materials Department's social committee, MAK, keeps track of special birthdays, anniversaries, weddings etc. and organizes annual events including a Christmas party and a summer picnic. The traditional Christmas party takes place in Roskilde and for the summer picnic, a scenic location, like the beaches of northern Zealand or the forests to the north of Copenhagen, is chosen.

For those interested in sports there are active groups in badminton, football, jogging, petanque, orienteering and summer groups of touring cyclists and rowers on the fjord. There is an annual ten-pin bowling tournament organized by MAK.

Members of the Materials Department are also active participants in hobby activities, organized through evening classes at Riso, including stone masonry, ornithology, piano classes and language training. There is a Riso art society which sponsors regular exhibitions in the foyer of the Riso canteen and loans out pictures to members' offices and to departments. There are also regular group trips organized by the organization 'Culture at work' to the theatre, museums and cultural events including evening lectures held at Riso by celebrities and artists. Furthermore, the various cultural activities of Roskilde and Copenhagen are easily accessible and offer lots of opportunities for visitors to participate in cultural events.

These Departmental social activities promote interactions between the regular staff and the increasing numbers of new graduate students, post docs and visiting scientists, many of them from abroad.

Participation in committees

The Materials Department is widely represented in scientific, educational and technical committees within Denmark and abroad. Such participation provides a strong base for international collaborative research activities and information exchange.

Danish committees

Technical Assessors, DANAK, Copenhagen.
Eivind Adolph and Niels Hansen

The Steering Committee for the Danish Solid Oxide Fuel Cell Programme, Riso.
Carsten Bagger, Niels Hansen, Per Knudsen and Mogens Mogensen

The Executive Committee of the Danish Society for Materials Research and Testing.
Janet J. Bentzen

Editorial Board of 'Keramisk Information'.
Janet J. Bentzen

The Executive Committee of the Danish Metallurgical Society, Lyngby.
Povl Brøndsted

The Board of Governors of Riso National Laboratory, Roskilde.
Morten Eldrup and Jens Olsson (Staff representatives)

The Danish Ministry of Energy, Advisory Group for Fuel Cells, Copenhagen.
Niels Hansen

The Steering Committee for the Centre for Advanced Technical Ceramics, Copenhagen.
Niels Hansen

Reference Group for the BRIT/ECRAM Programme, The Danish Ministry of Industry and Coordination, Copenhagen.
Niels Hansen

Ad Hoc Group on EC's Framework Programmes and Danish Research, The Danish Academy of Technical Sciences.
Niels Hansen

The Advisory Committee for the Engineering Science Centre (at Risø) for Structural Characterization and Modelling of Materials.

Niels Hansen and Dorte Juul Jensen

The Steering Committee for a Framework Programme on Advanced Thermoplastic Composites, Copenhagen.

Niels Hansen and Aage Lystrup

The Danish Electrical Research Institute (DEIRI), Lyngby.

Per Knudsen

The Danish Technical Research Council, Copenhagen.

Torben Leffers (Vice Chairman)

The Chemistry and Materials Commission of the Danish Technical Research Council, Copenhagen.

Torben Leffers (Chairman)

The Joint Committee for Biological Materials of the Danish Research Councils, Copenhagen.

Torben Leffers (Vice Chairman)

Task Force for the EC Programme on Standards, Measurements and Testing, Copenhagen.

Torben Leffers

The Research Committee of the Danish Society of Chemical, Civil, Electrical and Mechanical Engineers, Copenhagen.

Torben Leffers (Chairman)

Adjudication Committee for the Dr. Techn. Degree, Aalborg University.

Torben Leffers

The Steering Committee for the Centre for Polymer Composites, Copenhagen.

Hans Lilholt

The Advisory Committee for Continuing Education in Materials Technology, Copenhagen.

Aage Lystrup

The Executive Committee of the Danish Electrochemical Society, Copenhagen.

Mogens Mogensen

Steering Committee for Development and Characterization of Magnetic Materials, Danish Agency for Development of Trade and Industry, Copenhagen.

Allan Schröder Pedersen

Reference Group of Industry and Materials Technology, Danish Agency for Development of Trade and Industry, Ministry of Industry, Copenhagen.

Ole Toft Sørensen

International committees

Editorial Board of 'Journal of Strain Analysis'.

Svend Ib Andersen

The International Scientific Committee for the Second International Conference on Composite Testing and

Standardization, Hamburg, Germany.

Poul Brøndsted

The European Structural Integrity Society, Delft, The Netherlands.

Christian P. Debel

The International Advisory Committee on International Conferences on Positron Annihilation.

Morten Eldrup

Evaluation Group for the Joint Research Centre's Institute for Transuranium Elements, Karlsruhe, Germany.

Niels Hansen (Chairman)

The COST 501 Management Committee on Materials for Energy Conversion Using Fossil Fuels, Brussels, Belgium.

Niels Hansen

Editorial Board of 'Revue de Metallurgie'.

Niels Hansen

Editorial Board of 'Monographs in Materials Science'.

Niels Hansen

The Fusion Technology Steering Committee (FTSC-1), Brussels, Belgium.

Niels Hansen

International Organizing Committee for Fifth International Symposium on Plasticity and its Current Applications, Osaka, Japan.

Torben Leffers

Editorial board of 'Textures and Microstructures'.

Torben Leffers

Editorial Board of 'Plasticity'.

Torben Leffers

International Committee for Composite Materials, Philadelphia, USA.
Hans Lilholt

International Advisory Committee of the Tenth International Conference on Composite Materials, (ICCM-10), Vancouver, Canada.
Hans Lilholt

European Association for Composite Materials, Standing Committee.
Hans Lilholt

Editorial Board of 'Advanced Composite Materials'.
Hans Lilholt

Editorial Board of 'Composite Science and Technology'.
Hans Lilholt

Editorial Board of 'Advanced Composite Letters'.
Hans Lilholt

Editorial Board of 'Polymers and Polymer Composites'.
Hans Lilholt

Editorial Board of 'Applied Composite Materials'.
Hans Lilholt

Scientific Programme Committee of the Ninth International Conference on Mechanics of Composite Materials (CMCM), Riga, Latvia.
Hans Lilholt - Co-Chairman

The Nomination Committee for the Outstanding Achievement Award of the High Temperature Materials Division of the Electrochemical Society, Pennington, USA.
Mogens Mogensen

European Neutron Radiography Work Group.
Jorgen Rheimlinder

Nordic Collaboration Group for Storage and Conversion of Energy, Nordic Council of Ministers.
Allan Schröder Pedersen

The Fuel Cell Committee under the Nordic Energy Research Programme.
Finn W. Poulsen

International Energy Agency (IEA) Expert Group on SOFC.
Finn W. Poulsen

Organizing Committee for the First European Solid Oxide Fuel Cell Forum, Luzern, Switzerland.
Finn W. Poulsen

Expert Group on Structural Materials, EU Fusion Technology Programme, Brussels, Belgium.
Bachu N. Singh

Task Force Materials, EU Technology Programme, Brussels, Belgium.
Bachu N. Singh

Organizing Committee for Seventeenth International ASTM Symposium on Effects of Radiation on Materials, Sun Valley, USA.
Bachu N. Singh

International Organization Committee of the International Workshop on Assessment of Fundamental Aspects of Radiation Damage Production and Accumulation in Metals and Alloys, Obninsk, Russia.
Bachu N. Singh - Co-Chairman

Organizing Committee for the Symposium on 20 Years of Progress in Fusion Materials Research, Ispra, Italy.
Bachu N. Singh

International Organizing Committee for Ishino Conference on Fundamentals of Radiation Damage and Challenges for Future Nuclear Materials, Tokyo, Japan.
Bachu N. Singh

Nordic Society for Thermal Analysis and Calorimetry.
Ole Toft Sørensen

Standardization Committee, International Confederation for Thermal Analysis.
Ole Toft Sørensen

Published work

International publications

1. Afanas'ev, A.M.; Hendriksen, P.V.; Morup, S., Influence of rotational diffusion of the Mossbauer spectrum of ultra-fine particles in a supercooled liquid. *Hyperfine Interac.* (1994) v. 88 p. 35-48
2. Ahlgren, E.; Poulsen, F.W., Thermoelectric power of YSZ. In: Solid state ionics-93, Part 1, 9. International conference on solid state ionics, The Hague (NL), 12-17 Sep 1993. Boukamp, B.A.; Bouwmeester, H.J.M.; Burgeraaf, A.J.; Put, P.J. van der; Schoonman, J. (eds.). (North-Holland, Amsterdam, 1994) (Solid State Ionics, 70/71) p. 528-532
3. Ahlgren, E.O., Thermoelectric power of solid oxide fuel cell materials. *Riso-R-762* (N) (1994) 154 p
4. Ahlgren, E.O.; Ramløv, J.; Poulsen, F.W., On the thermoelectric power of a mixed ionic-electronic conductor. In: 2nd Nordic symposium on high temperature fuel cells. Proceedings, 2. Nordic symposium on high temperature fuel cells, Geilo (NO), 16-18 Mar 1994. Norby, T.; Poulsen, F.W. (eds.). (Nordisk Energiforskningsprogram, Energiforskningen, Ås, 1994) (Nordisk energiforskningsamarbejde) p. 221-226
5. Ahlgren, E.O.; Ramløv, J.; Poulsen, F.W., Thermoelectric power of rare earth aluminates. In: Ionic and mixed conducting ceramics. Proceedings, 2. International symposium on ionic and mixed conducting ceramics, San Francisco, CA (US), 22-27 May 1994. Ramanarayanan, T.A.; Worrell, W.L.; Tuller, H.L. (eds.). (The Electrochemical Society, Pennington, NJ, 1994) (High Temperature Materials Division Proceedings Volume, 94-12) p. 598-607
6. Albertini, G.; Cernuschi, E.M.; Cicognani, G.; Gibu, S.; Lorentzon, T.; Rustichelli, F., Residual strain measurements in welded steel Fe-10Cr. In: Proceedings of the fourth international conference on residual stresses, 4. International conference on residual stresses, 1994, 4. Baltimore, MD (US), 8-10 Jun 1994. (Society for Experimental Mechanics, Bethel, CT, 1994) p. 969-973
7. Anderson, S.I.; Bakke-Sørensen, J.B.; Lorentzon, T.; Pedersen, O.B.; Sørensen, V.J. (eds.), Numerical predictions of deformation processes and the behaviour of real materials. Proceedings, 15. Riso international symposium on materials science, Riso (DK), 5-9 Sep 1994. (Riso National Laboratory, Roskilde, 1994) 644 p.
8. Anderson, S.I.; Lilholt, H.; Lystrup, A., Fatigue properties and design of wing blades for wind turbines. Contract no. 9010-0071-DK (1992). Fatigue diagrams for glass/polyester composite materials. Final report for the period April 1, 1990 to March 31, 1993. (Riso National Laboratory, Materials Department, Roskilde, 1994) 1 p.
9. Bøggert, C.; Hendriksen, P.V.; Mogensen, M., Experimental studies on stacking of planar SOFCs. In: First European solid oxide fuel cell forum. Proceedings, Vol. 2, 1. European solid oxide fuel cell forum, Lucerne (CH), 3-7 Oct 1994. Bessel, U. (ed.). (European SOFC Forum Secretariat, Baden, 1994) p. 691-702
10. Bøggert, C.; Mogensen, M.; Walker, C.T., Temperature measurements in high burn-up UO₂ nuclear fuel. Implications for thermal conductivity, grain growth and gas release. *J. Nucl. Mater.* (1994) v. 211 p. 11-29
11. Bay, N.; Berregaard, H.; Petersen, S.B.; Santos, C.H.G. dos, Cross shear roll bonding. *J. Mater. Process. Technol.* (1994) v. 45 p. 1-6

12. *Rutten, J.J.* Sandwich sensors. In: Centre for Advanced Technical Ceramics Final report May 1989 - April 1994. Toft Sørensen, O. (ed.). (Riso National Laboratory, Materials Department, Roskilde, 1994) p. 239-245.
13. *Rutten, J.J.; Kwon, H.* Development and testing of ceramic oxygen sensors with improved thermal shock and corrosion resistance. In: Centre for Advanced Technical Ceramics Final report May 1989 - April 1994. Toft Sørensen, O. (ed.). (Riso National Laboratory, Materials Department, Roskilde, 1994) p. 135-151.
14. *Bilde-Sørensen, J.B.; Smith, D.A.* Comment on: 'Retardation of the relationship between denuded zones and diffusional creep'. *Ser. Metall. Mater.* (1994) 3, 3, p. 383-386.
15. *Bjerrgaard, H.; Andersen, C.B.; Jøel Jensen, D.; Wanhem, T.* Flange thickness in a radial extruded tubular component - strain state, texture and strain models. In: Numerical predictions of deformation processes and the behaviour of real materials. *Proceedings 15. Riso international symposium on materials science*, Riso (1994), 5-9 Sep 1994. Andersen, S.L.; Bilde-Sørensen, J.B.; Lorenzen, E.; Pedersen, O.B.; Sørensen, N.J. (eds.). (Riso National Laboratory, Roskilde, 1994) p. 249-254.
16. *Bonanos, A.* Electrochemical studies of methane oxidation on silver, platinum and nickel/nickel. In: 2nd Nordic symposium on high temperature fuel cells. *Proceedings 2. Nordic symposium on high temperature fuel cells*, Guelo (1994), 16-18 Mar 1994. Norby, T.; Poulsen, F.W. (eds.). (Nordisk Energiforskningsprogram, Energiforskningen, Ås, 1994) (Nordisk energiforsknings-samarbejde) p. 169-174.
17. *Bromberg, P.; Andersen, S.L.* Measurement of the compression properties of CTR punches with delaminations and repaired impact damages. In: Composites testing and standardisation 2. European conference on composites testing and standardisation, ICUMC 18.2, Hamburg (1994), 13-15 Sep 1994. Hogg, P.J.; Schulte, K.; Wittich, H. (eds.). (Woodhead Publishing Limited, Abingdon, 1994) p. 159-166.
18. *Bromberg, P.; Heredia, F.E.; Evans, A.G.* In-plane shear properties of 2-0 ceramic matrix composites. *J. Am. Cer. Soc.* (1994) 77, p. 2569-2574.
19. *Bromberg, P.; Johansen, B.S.; Mægaard, V.; Sørensen, P.S.* Simulation of an extrusion process by tension-torsion testing. In: Numerical predictions of deformation processes and the behaviour of real materials. *Proceedings 15. Riso international symposium on materials science*, Riso (1994), 5-9 Sep 1994. Andersen, S.L.; Bilde-Sørensen, J.B.; Lorenzen, E.; Pedersen, O.B.; Sørensen, N.J. (eds.). (Riso National Laboratory, Roskilde, 1994) p. 255-261.
20. *Bodker, E.; Morup, S.; Linderoth, S.* Surface effects in metallic iron nanoparticles. *Phys. Rev. Lett.* (1994) 72, p. 252-255.
21. *Bodker, E.; Morup, S.; Linderoth, S.* Influence of chemisorption on the magnetic properties of metallic iron nanoparticles. In: International conference on magnetism. Programme and abstracts. International conference on magnetism, 18-20 Aug 1994, Warsaw (PL), 22-26 Aug 1994. Krömpiewski, S.; Szajek, A.; Morkowski, J. (eds.). (Ośrodek Wydawnictw Naukowych, Poznań, 1994) p. 304.
22. *Carlson, H.* Decommissioning of the Riso Hot Cell facility - 7. Periodic report covering July 1 to December 31, 1993. Riso-Hot-Decom-P-7 (1994) 7 p.
23. *Carlson, H.* Decommissioning of the Danish Hot Cell Facility at Riso. In: 1994 International symposium on decontamination and decommissioning. *Proceedings 1994 International symposium on decontamination and decommissioning*, Knoxville, TN (USA), 25-28 Apr 1994. Underwood, P.; Collins, K. (eds.). (1994-940306 (1994) Session H.
24. *Carlson, H.* Decommissioning of the Riso Hot Cell facility. Final Report. (Riso National Laboratory, Materials Department, Hot Cell Facility, Roskilde, 1994) (Riso-Hot-Decom-Final) 40 p.
25. *Carter, J.D.* Sinterability and reaction stability of lanthanum chromites. In: 2nd Nordic symposium on high temperature fuel cells. *Proceedings 2. Nordic symposium on high temperature fuel cells*, Guelo (1994), 16-18 Mar 1994. Norby, T.; Poulsen, F.W. (eds.). (Nordisk Energiforskningsprogram, Energiforskningen, Ås, 1994) (Nordisk energiforsknings-samarbejde) p. 197-202.
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Abstract (Max. 2000 characters)

The annual report describes the work of the Materials Department at Riso National Laboratory during 1994. The work is presented in three main chapters: Materials Science, Materials Engineering and Materials Technology. The report includes lists of staff members, guests, post docs and PhD students. There are detailed lists of the published work which has resulted from the projects.

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RISO NATIONAL LABORATORY is a broad-based research organization with primary research activities in energy, the environment and in materials. There is a total of 949 employees.

The research programmes of the Materials Department include basic studies of materials structure and properties, structural mechanics and materials testing, and processing techniques for polymer composites, powder metallurgical products and engineering ceramics. Advanced characterization techniques used in the Department are electron microscopy, positron annihilation, neutron diffraction, small angle neutron scattering and mechanical testing.

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